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INGENIEROS DE CAMINOS, CANALES Y PUERTOS

**High Strain Rate Mechanical Behavior of
Advanced High Strength Steels**

Doctoral Thesis

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To find yourself, think for yourself.

Socrates

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Resumen

Los requisitos de aligeramiento y optimización de la eficiencia del combustible de los vehículos promovieron el desarrollo de aceros avanzados de alta resistencia (AHSS), que se caracterizan por su alta resistencia y ductilidad. Hasta ahora, se han propuesto tres generaciones de AHSS y se han llevado a cabo importantes actividades de investigación sobre ellos. La gran mayoría de estos estudios se centró en el diseño microestructural para mejorar las propiedades mecánicas de tracción básicas de los AHSS. Sin embargo, su comportamiento a altas tasas de deformación apenas se ha estudiado, a pesar de su relevancia significativa para aplicaciones automovilísticas. Esta tesis se centra en la respuesta de tres AHSS a la carga dinámica. Son acero de doble fase (DP) 1180, acero inoxidable 304 (304 SS) y acero de temple y particionado (Q&P), pertenecientes a los AHSS de primera, segunda y tercera generación, respectivamente.

La respuesta mecánica, la resistencia al impacto, la evolución de la microestructura tras el impacto y el calentamiento adiabático se estudiaron a fondo. Se utilizó el sistema de prueba por impacto de caída de peso para la prueba de impacto en las hojas AHSS de 1 mm de espesor. El aumento de temperatura inducido por el calentamiento adiabático se midió cuantitativamente in situ durante la deformación a altas velocidades de deformación. La evolución de la microestructura se caracterizó ampliamente por técnicas de difracción de retrodispersión de electrones (EBSD) y microscopía electrónica de transmisión (TEM). Los resultados experimentales indicaron que, en las condiciones actuales, 304 SS tiene la mejor resistencia al impacto (130 J), seguido del acero Q&P (110 J) y el acero DP (90 J). La resistencia al impacto del acero Q&P se ve reforzada por el efecto combinado del deslizamiento de dislocaciones y el fenómeno TRIP. Estos mecanismos junto con el mecanismo de hermanamiento aumentan aún más la resistencia al impacto de 304 SS. Debido a la combinación de excelentes propiedades mecánicas y bajo costo, el acero Q&P es más atractivo que los aceros 304 SS y DP para aplicaciones automovilísticas.

Además, la respuesta del acero Q&P se examinó cuidadosamente en una amplia gama de tasas de deformación (10^{-4} - 10^3 s $^{-1}$) mediante el uso de una máquina universal de ensayos, un sistema de barra de tensión dividida Hopkinson (SHTB) y una técnica de correlación de imagen digital (DIC). El límite elástico (YS) del acero Q&P en pruebas dinámicas (500 - 1000 s $^{-1}$) es 200 MPa más alto en comparación con las pruebas estáticas (10^{-4} y 10^{-2} s $^{-1}$), mientras que la resistencia a la tracción final (UTS) tiende a aumentar linealmente con la velocidad de deformación. La fracción de austenita retenida (RA) disminuye exponencialmente con el aumento de la deformación plástica durante las pruebas de tracción estáticas y dinámicas.

Abstract

The requirements of lightweighting and optimizing fuel efficiency of vehicles promoted the development of advanced high strength steels (AHSSs), which are characterized by high strength and ductility. Until now, three generations of AHSSs have been developed, and significant body of research on this topic exists in the current literature. The vast majority of studies focused on the microstructural design to improve basic tensile mechanical properties of AHSSs. However, their dynamic behavior and impact resistance have not been systematically investigated, despite their significant relevance for automotive applications. This thesis focuses on the high strain rate performance of three AHSSs. These are a dual phase (DP) 1180 steel, a 304 stainless steel (304 SS) and a quenching and partitioning (Q&P) steel, belonging to the first, second and third generation AHSSs, respectively.

The main emphasis of this experimental work was laid on the impact resistance of the 1 mm thick AHSSs sheets subjected to drop weight impact testing. Mechanical behavior, microstructure evolution and mechanisms operating during high strain rate deformation were analyzed. Microstructure evolution was comprehensively characterized by electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM) techniques. Special attention was paid to adiabatic heating (which was measured *in situ*) and its interplay with the deformation mechanisms. The experimental results indicated that the 304 SS has the best impact resistance (130 J), followed by the Q&P steel (110 J) and the DP 1180 steel (90 J). The impact resistance of the Q&P steel was enhanced by the combined effect of dislocation glide and TRIP phenomenon. These mechanisms along with twinning mechanism further increased the impact resistance of the 304 SS. Due to the combination of excellent mechanical properties and low cost, the Q&P steel appears to be more attractive for automotive application compared to the 304 SS and DP steel.

Additionally, tensile mechanical behavior of the Q&P steel was carefully examined in

a wide range of strain rates (10^{-4} – 10^3 s $^{-1}$) by employing split Hopkinson tensile bar (SHTB) system and digital image correlation (DIC) technique. It was shown that the yield strength (YS) of the Q&P steel in dynamic tests (500 – 1000 s $^{-1}$) was by 200 MPa higher compared to the static tests (10^{-4} and 10^{-2} s $^{-1}$), while the ultimate tensile strength (UTS) tended to increase linearly with strain rate. Retained austenite (RA) fraction decreased exponentially with the increase of plastic strain during both static and dynamic tensile testing. Comparative analysis of the outcomes of SHTB and drop weight impact testing showed that high strain rate biaxial deformation promoted transformation of retained austenite into martensite along with the ductility of the Q&P steel.

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List of Abbreviations

AHSSs	advanced high strength steels
AISI	American iron and steel institute
ASTM	American society for testing and materials
AUST. SS	austenitic stainless steel
bcc	body centered cubic
BF	bright field
BSE	backscattered electrons
CBED	convergent beam electron diffraction
CP	complex phase
D&P	deformed and partitioned
DARA	dislocation absorption of retained austenite
DBTT	ductile-brittle transformation temperature
DEM	digital elevation model
DENT	double edge-notched tension
DF	dark field
DIC	digital image correlation
DP	dual phase
DSA	dynamic strain aging
EBSD	electron backscatter diffraction
ECCI	electron channeling contrast imaging
EDS	energy dispersive spectrum
EL _t	total elongation
EL _u	uniform elongation
fcc	face centered cubic
FEG	field emission gun
FEM	finite element method
FFT	fast Fourier transformation
FIB	focused ion beam
FLC	forming limit curve
HAB	high angle boundary
HCF	high-cycle fatigue
HRTEM	high resolution transmission electron microscopy

List of Abbreviations

HSLA	high strength low alloy
IPF	inverse pole figure
IQ	image quality
KAM	kernel average misorientation
K-S	Kurdjumov-Sachs
LCF	low-cycle fatigue
L-IP	light weight steel with induced plasticity
MART	martensitic
MBIP	micro band induced plasticity
MTSC	maximum tensile stress criterion
NSSD	normalized sum of squared differences
Q&P	quenching and partitioning
QAT	quenching and austempering
QT	quenching and tempering
RA	retained austenite
RD	rolling direction
SAED	selected area electron diffraction
SBIP	shear band induced plasticity
SEM	scanning electron microscope
SFE	stacking fault energy
SHTB	split Hopkinson tensile bar
SIMT	strain induced martensite transformation
S-N	stress amplitude vs. number of cycles to fatigue failure
STEM	scanning transmission electron microscopy
TD	transverse direction
TEM	transmission electron microscopy
TKD	transmission Kikuchi diffraction
TM	tempered martensite
TMP	thermomechanical processing
TRIP	transformation induced plasticity
TWIP	twinning induced plasticity
ULSAB	ultralight steel auto body
UM	untempered martensite
UTS	ultimate tensile strength
XRD	X-ray diffraction
YS	yield strength

Chapter 1

Introduction

Increasing passenger safety and simultaneously decreasing vehicle weight are the two primary objectives for automotive manufacturing industry. This exploration has not been interrupted since the advent of automobiles. Until now, a tremendous number of unique materials, including steels, aluminum alloys, plastic, carbon fiber, ceramics, composites, etc., have already been developed and applied to different parts of automobiles to obtain upper security level, less body weight and higher fuel efficiency. Although substantial load-carrying or ornamental components are constructed without evolving steel, steel remains the most important material for most cars at now. In a 1400 kg passenger car, which represents the prevailing gross at present, steel components and units account for more than 65% of the whole body weight [1,2], as can be seen in Fig. 1.1. In the foreseeable future, the percentage occupied by steel in a car weight will proceed to increase, due to wider application of Advanced High Strength Steels (AHSSs) [3]. Consequently, to realize weight reduction for automobiles, i.e., lightweighting, consideration should be intentionally focused on those parts shaped by steel.

1.1 Advanced high strength steels (AHSSs)

Fig. 1.1 Material distribution of vehicle curb weight in kg [1,2].

To get passenger car weight loosen and fuel efficiency increased, resorting to AHSSs is one of the most promising and competent approaches. Compared to the conventional high strength steels, in which ductility is reduced with increasing strength, AHSSs are characterized by combination of high strength, good formability and crashworthiness. Based on tensile mechanical strength, the AHSSs are roughly referred to those with yield strength (YS) >300 MPa and ultimate tensile strength (UTS) > 600 MPa [4]. Started since late 1980s with the investigation on dual phase (DP) steel [5], three generations of AHSSs have been established according to the development stages [6-8]: 1) the first generation AHSSs, i.e., DP, CP (complex phase), MART (martensitic) and TRIP (transformation induced plasticity) steels; 2) the second generation AHSSs, including TWIP (twinning induced plasticity), AUST. SS (austenitic stainless steel) and L-IP (light weight steel with induced plasticity) steels; 3) the third generation AHSSs including Q&P (quenching and partitioning) and medium Mn steels. Ashby plot showing tensile elongation - ultimate tensile strength relationships for various AHSSs is presented in Fig. 1.2. It is seen that the second generation AHSSs have better ductility than the first generation AHSSs, while the third generation AHSSs occupy a wide range in the elongation - strength map and partially fill up the property gap between the second and the first generation AHSSs.

Fig. 1.2 Ashby plot of tensile elongation vs. ultimate tensile strength for various AHSSs [3].

1.1 Advanced high strength steels (AHSSs)

In the past few decades, by adopting novel alloy composition design and new thermomechanical processing technologies together with unique strengthening mechanisms, excellent AHSSs have been developed inside laboratories or even applied to auto bodies [4,9–11]. These steels possess high strength, good formability and crashworthiness. According to the guidelines issued by WorldAutoSteel, an international union comprised of 21 major global steel producers, the use of AHSSs in automobiles has been rapidly growing [3], as can be seen in Fig. 1.3. Apparently, AHSSs will be the prevailing material used in vehicle bodies during next decades. Below, each generation of AHSSs is briefly profiled.

Fig. 1.3 The rapid growth of AHSSs used in automobiles [3].

1.1 Advanced high strength steels (AHSSs)

1.1.1 First generation AHSSs

The main motivation of developing the first generation AHSSs was to obtain higher strength compared to the conventional high strength steels by adopting similar chemical composition but without unaffordable cost increase [6]. As can be seen in Table 1.1, the first generation AHSSs are characterized by relatively low content of alloying elements and up to four microconstituents present in microstructure. Some of these steels have been applied in automotive industry with increasing proportion, as shown in Fig. 1.3. They have been continuously improved by academic community. Most widely used first generation AHSSs include DP steel, TRIP steel, CP steel and MART steel.

Table 1.1 The microstructures and chemical compositions of the first generation AHSSs reported in literature. (A: austenite; B: bainite; F: ferrite; M: martensite; RA: retained austenite)

Steel	Microstructure	Chemical composition (wt.%)	Ref.
DP	F + M	Fe-0.07C-1.42Mn-0.83Si	[12]
		Fe-(0.10~0.19)C-(1.43~1.54)Mn-(1.93~2.09)Si	[13]
		Fe-(0.034~0.23)C-(0.22~0.36)Mn-(0.05~0.06)Al	[14]
	F + M + RA	Fe-(0.034~0.23)C-(0.22~0.36)Mn-(0.01~0.02)Si	[15]
	F + M + carbide	Fe-(0.07~0.11)C-(1.52~2.67)Mn-(0.02~0.25)Si	[16]
	F + M + RA + carbide	Fe-0.06C-1.62Mn-0.23Si	[17]
TRIP	F + M + RA + B	Fe-(0.19~0.31)C-1.57Mn-(0.34~1.46)Si	[18]
		Fe-0.18C-2.45Mn-1.03Si	[19]
		Fe-(0.10~0.14)C-1.50Mn-1.48Si	[20]
		Fe-0.20C-1.80Mn-2.00Si	[21]
CP	F+M + RA + B	Fe-0.14C-2.5Mn-0.14Si	[22]
		Fe-(~0.15)C-(1.50~2.5)Mn-(<0.50)Si-(<0.70)Cr	[23]
	F + M + B	Fe-0.11C-2.85Mn-0.10Si	[24]
	M + RA + B	Fe-0.15C-2.2Mn-0.05Si	[25]
MART	M	Fe-0.03C-0.3Mn	[26]
	M + carbide	Fe-0.3C-1.00Mn-0.29Si-0.50Cr-0.02Nb	[27]

The starting point of developing the first generation AHSSs was to meet the requirement of having better strength and ductility but just at slightly higher cost than conventional high strength steels, to deal with the increasing fuel crisis and greenhouse gases emission problem. The first member of AHSSs family is DP steel, even though the

concept of AHSS did not exist when DP steel appeared in 1980s [5]. Typical microstructure of DP steels consists of soft ferrite and hard martensite, as shown in Fig. 1.4. Martensite has irregular shape and random distribution in ferritic matrix. Small fraction of carbide and/or retained austenite (RA) may also exist in DP steels [15–17]. The frequently added alloying elements in DP steels are manganese (Mn) and silicon (Si), composing the basic Fe–C–Mn–Si system. Mn is used to stabilize austenite and Si to promote austenite-ferrite transformation [6]. The carbon content has significant effect on the UTS of DP steels [13], due to its solid solution strengthening effect on martensite. The strength of DP steels is mainly provided by martensitic phase while the ductility by ferrite. This composite-like structure at microscale enables DP steels to reach UTS in the range of 500~1200 MPa [13,14]. It is widely accepted that the relationship between the martensite fraction and tensile strength of DP steels is linear, namely, the higher ratio of martensite leads to the higher strength [13,16]. However, the strength improvement of DP steels by simply increasing the martensite fraction degrades their formability, resulting in limited hole expansion capability [3]. One solution is to refine the microstructure of DP steels, for instance, via thermomechanical processing (TMP) [17].

Fig. 1.4 An example of the microstructure of DP steel (Optical microscopy, the white is martensite and the rest is ferrite) [12].

1.1 Advanced high strength steels (AHSSs)

Another outstanding member of the first generation AHSSs group is TRIP steel. The development of steels with so-called TRIP effect dates back to the report by V.F. Zackay *et al.* in 1967 [28]. The most prominent feature of these steels is the promotion in ductility with the assistance of martensitic transformation of metastable RA under external loading. On microscale, RA, which has a minimum volume fraction of 5%, is embedded inside the ferritic matrix of TRIP steels [3,6]. Other phases with varying fraction also exist in TRIP steels, e.g., bainite and martensite. A typical microstructure of TRIP steel is schematically shown in Fig. 1.5. Such multiphase microstructure is usually achieved by controlled cooling after intercritical annealing, and then isothermally holding in bainite transformation region (austempering) followed by quenching to room temperature [6]. During intercritical annealing, a mixture of ferrite and austenite formed while carbon atoms accumulated into austenite, which is the first carbon enrichment process. However, the carbon content in austenite after this process is not high enough to retain the austenite at room temperature. Therefore, the second carbon enrichment is performed during bainite reaction treatment, where part of austenite is partitioned with enough carbon atoms to be remained during the final quenching as its martensite transformation temperature (M_s) is lower than room temperature. Along with Mn, another conventional alloying element, Si, is added into TRIP steels to obtain the desired microstructure [4]. However, the addition of Si above ~0.3% degrades galvanizability of steel [29]. So Si is partially or completely substituted by Al. TRIP steels based on C-Mn-Al system have been the prevailing direction for further industry's choice [18].

Fig. 1.5 Schematic drawing of the microconstituents in TRIP steels [3].

Complex phase (CP) steel is a steel grade with tensile strength of ≥ 800 MPa, consisting of ferrite/bainite matrix and minor fraction of RA, martensite and/or pearlite [3,6]. Fig. 1.6a shows the microstructure of a CP 800 steel after color etching with Le-Pera agent while Fig. 1.6b displays the microstructural features of bainite region under SEM. Typically, CP steels have similar chemical compositions as DP and TRIP steels, but with small additions of Niobium (Nb), Titanium (Ti) and Vanadium (V) mainly to obtain the precipitate strengthening effect [4,6,30]. Additionally, Nb or Ti have excellent grain refining effect on the microstructure of CP steels. The strength and ductility of CP steels can be varied in a wide range by tuning the ratio of different phases. Except RA, the fraction of ferrite/bainite, martensite can be adjusted by controlling the cooling rate after austenitization [6,31]. Furthermore, by means of complex microstructure, CP steels have remarkable capabilities in energy absorption, formability and hole expansion, all of which are desirable for automotive application. For instance, the comparative study done by A. Karelova *et al.* [22] indicated that the CP 800 steel exhibited superior performance in hole expansion testing when compared with DP 800 steel.

Fig. 1.6 Optical microscope image of a CP 800 steel: ferrite (blue), bainite (rust color) and martensite (yellow). (2) SEM image of bainite region. Carbides and martensite/austenite (MA) islands are indicated by white arrows [32].

The name of Martensitic (MART) steels refers to the main phase in these steels, although some ferrite and/or bainite with small amount may be present as well [3,6]. The typical microstructure of MART steels is shown in Fig. 1.7. The martensite structure forms due to quenching of austenite after hot-rolling or annealing. The post-quenching tempering is frequently carried out to achieve good formability with ultrahigh strength. Carbon is the most critical alloying element in MART steels due to its dominate role in controlling hardenability and strength [4]. MART steels are characterized by extraordinary strength, having UTS as high as 1700 MPa, or even higher [4,26,27].

Fig. 1.7 SEM image of a MART steel after nital etching [27].

A substantial progress has been achieved in the development of the first generation AHSSs by both laboratories and industry during past decades. Their application in auto

bodies (e.g., DP steel) is increasing year by year, as demonstrated in Fig. 1.3. The lean alloy strategy was employed by all steel manufacturers of the first generation AHSSs, which confines their cost from the start, and is attractive for car manufactures. The low ductility of the first generation AHSSs, which is their main drawback, was one of the reasons for the development of the second generation AHSSs.

1.1.2 Second generation AHSSs

The biggest disadvantage of the first generation AHSSs is their relatively low ductility (as shown in Fig. 1.2), as handling the dilemma between the strength and ductility is a well-known tough mission. The second generation AHSSs were explored to develop steels with excellent combination of high strength and ductility, thus enable the production of components where high formability is necessary. Table 1.2 summarizes the microstructures and chemical compositions of the second generation AHSSs. It is clear that high content of alloying elements, for instance manganese and/or aluminum, are added into these steels, which results in their higher cost compared to the first generation AHSSs [6]. TWIP and AUST. SS have only austenitic microstructure while L-IP steels possess also other phase(s). Three remarkable steels of the second generation AHSSs group, i.e., TWIP, AUST. SS and L-IP steels, are shortly overviewed below.

Table 1.2 The microstructures and chemical compositions of the second generation AHSSs reported in literature. κ -carbide is (Fe, Mn) AlC_{1-x} [33] while B2 phase corresponds to (Fe,Ni)Al [34]. (A: austenite; F: ferrite)

Steel	Microstructure	Chemical composition (wt.%)	Ref.
TWIP	A	Fe-(0.52~0.55)C-(12.1~12.5)Mn-(0/3.6)Al-(0/4.8)Ni	[35]
		Fe-0.4C-17Mn-0.06V	[36]
		Fe-0.6C-20Mn	[37]
		Fe-0.6C-15Mn-1.2Al	[38]
		Fe-0.7C-15Mn-2Si-2Al	[39]
AUST. SS	A	Fe-(0.02/0.08)C-7.1Mn-(16.3~16.9)Cr-(4.5~5.4)Ni-0.5Si	[40]
		Fe-(0.02~0.08)C-(0.8~1.8)Mn-(16.4~18.8)Cr-(6.8~12.1)Ni-0.5Si	[40]
L-IP	A + F + κ -carbide	Fe-(0.7~1.2)C-(18~28)Mn-(9~12)Al	[33]
	A + F	Fe-0.3C-8.5Mn-5.6Al	[41]
	A + κ -carbide	Fe-1.11C-29.8Mn-7.65Al-0.1Si	[42]
	A + b2 phase	Fe-(0.8/1.0)C-(15.0/16.0)Mn-(8.0/10.0)Al-5.0Ni	[34]

1.1 Advanced high strength steels (AHSSs)

The austenitic TWIP steels basically have a Fe-Mn-C composition system, where high amount of manganese (12~30 wt.%) and carbon (0.4~1.0 wt.%) are used to stabilize austenite at room temperature [35,36,43-45]. Various amount of silicon (Si) and/or aluminum (Al) may also be added to achieve the combination effect of retarding carbide precipitation, stabilizing austenite and solid solution strengthening [6,46,47]. In TWIP steels, the high strength, elongation and strain hardening exponent are realized through twinning deformation mechanism. The desired microstructure of TWIP steel is uniform and carbide free austenite, and minor fraction of martensite [48] and/or carbide [49] may appear in the microstructure as well. The excellent mechanical properties of TWIP steels are achieved through deformation of austenite grains via the development of dislocations, twins and martensite, depending on the strain path [48]. Dislocation glide is the prevailing deformation mode in TWIP steels [6,44]. Mechanical twins occur and thrive after a certain level of strain [48,50]. The continuously formed twin boundaries act as the barrier for dislocation movement, reducing the available glide distance of dislocations and strengthening the material, which is the so-called dynamic Hall-Petch effect [44,50]. The mechanical twins developed during deformation in TWIP steels are shown in Fig. 1.8, revealed by electron channeling contrast imaging (ECCI) technique [51]. Extensive twinning during plastic deformation of TWP steels is enabled by their reduced stacking fault energy (SFE), which is the energy per area of fault and can be regarded as a surface tension pulling the partial dislocations together [52]. The reported SFE range for twinning is roughly located in $18 \text{ mJ/m}^2 \leq \text{SFE} \leq 45 \text{ mJ/m}^2$, while phase transformation and dislocation gliding will occur when SFE is below and above this region [53]. It should be noted that both the critical values for activation of each deformation mechanism and the measured SFE of a material remain a matter of debate [54,55].

Fig. 1.8 ECCI images of deformed austenite grain at 0.3 true strain: (a) Large view field of twin structure; (b) details of the lamellar twin structure. The ECCI images were obtained by orienting the grain into Bragg condition using the $(111)g$ vector (arrows) [51].

AUST. SSs are characterized by their outstanding mechanical and corrosion resistance properties [6,7,56]. Large amount of expensive alloying elements, such as chromium (Cr, 16~25 wt.%) and nickel (Ni, 4~13 wt.%), are added into AUST. SSs to withstand external corrosive attack [40]. Chromium is an indispensable element in AUST. SSs to obtain sufficient corrosion resistance [6,40]. It also improves the resistance to high temperature oxidation. Nickel has a role in stabilizing austenite and enhancing corrosion and oxidation bearing capability. These elements are the source of the most obvious drawback of AUST. SSs, which is their very high cost. Therefore, investigations on reducing/eliminating nickel in AUST. SSs have been carried out in recent years [57,58]. Like TWIP steels, AUST. SSs have austenitic microstructure. However, AUST. SSs have lower SFE than that of TWIP steels, which falls in the overlapping SFE region where both twinning and phase transformation occur during deformation [55]. The twins and martensite phase developed during deformation in an AUST. SS are shown in Fig. 1.9 [59]. The most representative AUST. SSs are the 3XX series steel grades, which have wide application in nuclear reactor [60], food [61] and automobile industry [62].

Fig. 1.9 Phase maps of a Fe-0.006C-18.75Cr-11.7Ni (wt.%) AUST. SS after plastic deformation (*in situ*) to different strains: (a) 8.2% and (b) 18.2%. Austenite is in red, martensite in blue, deformation twins in yellow and HABs in black. Band contrast maps were used as background [59]. HAB: high angle boundary.

The most evident feature of L-IP (lightweight steel with induced plasticity) steels is their lower density than that of other AHSSs or conventional steels [63–65], which attracted a lot of attention from automobile manufacturers. The reduced density in L-IP steels is mainly realized by adding aluminum. For every 1 wt.% aluminum added in L-IP steels, 1.3% of density reduction can be reached [33]. While the downside is that aluminum deteriorates the Young's modulus and work hardening rate of L-IP steels [33,65]. High amount of manganese is also added to increase the stability of austenite. In L-IP steels, the content of manganese and aluminum have wide ranges: 8~28 wt.% and 5~12 wt.%, respectively [33,41]. Depending on the constituent phase, L-IP steels can be classified into three categories [6]: 1) austenitic, 2) ferritic and 3) “austenite+ferrite” duplex steels. Precipitates, such as κ -carbide [42] and B2 phase [34,66], may form in L-IP steels depending on the chemical composition and thermomechanical route. The deformation behavior and mechanisms of L-IP steels depend on their microstructure. The investigation on Fe-(26~28)Mn-(10~12)Al-(1.0~1.2)C steels revealed that the so-called shear band induced plasticity (SBIP) or micro band induced plasticity (MBIP) controlled the deformation process in the “austenite+ferrite” duplex L-IP steels due to the high SFE ($\approx 110 \text{ mJ/m}^2$) [33]. The TEM image of shear bands developed in a L-IP steel is shown in Fig. 1.10 [33]. The study on a Fe-0.3C-8.5Mn-5.6Al (wt.%) ferrite-austenite duplex lightweight steel showed that the excellent ductility (77%) can be achieved by combination

of TWIP and TRIP effects [41]. While only TWIP effect but no TRIP effect was observed in the Fe-30Mn-(0, 5, 8.5)Al-0.85C (wt.%) steels under loading [67].

Fig. 1.10 TEM bright-field image exhibits shear bands on {111} planes in the austenitic matrix [33].

Austenite is the main phase or matrix of the second generation AHSSs, which results in the 'soft' nature of these steels and is the root of their excellent ductility. Work hardening of austenite is the main approach to improve tensile strength of these steels. Simultaneously possessing high strength and high elongation is desirable for vehicle manufacturers. However, the wide application of the second generation AHSSs in automotive industry is confined due to their very high cost, which encouraged the development of the third generation AHSSs, which are described in the next section.

1.1.3 Third generation AHSSs

The third generation AHSSs were proposed with the objective to achieve a better combination of strength and ductility than the first generation AHSSs and affordable price for consumers [3]. They are characterized by limited alloy addition to control the cost to the level slightly higher than that of the first but much lower than that of the second generation AHSSs [6]. Their mechanical properties fill up the gap between the first and second generations of AHSSs (Fig. 1.2). The exploration on the third generation AHSSs is ongoing and many conceptions and definitions are not unified yet [7,8,11]. As can be seen in Table 1.3, the third generation AHSSs have typically two or three phases in

1.1 Advanced high strength steels (AHSSs)

microstructure and limited alloy addition. Two promising candidates of the third generation AHSSs, i.e., Q&P steel and medium Mn steel, are profiled below.

Table 1.3 The microstructures and chemical compositions of the third generation AHSSs reported in literature. (A: austenite; F: ferrite; M: martensite; RA: retained austenite)

Steel	Microstructure	Chemical composition (wt.%)	Ref.
Q&P	M ¹ + RA	Fe-(0.025/0.28)C-(2.5/3.0)Mn-(1.5/2.0)Si-(0.02/0.15)Cr	[68]
		Fe-0.21C-0.29Mn-1.75Si-1.03Cr-2.86Ni-0.31Mo-0.05Nb	[69]
	M + RA + carbide	Fe-(0.20/0.30)C-(3.0/5.0)Mn-1.6Si	[70]
		Fe-(0.2/0.3)C-4.0Mn-1.6Si-1.0Cr	[71]
	M + F ² + RA	Fe-0.19C-1.61Mn-0.35Si-1.1Al	[72]
Medium Mn	F + RA	Fe-0.1C-7.0Mn-0.5Si	[73]
	F + M + RA	Fe-0.09C-4.6Mn	[74]
		Fe-0.05C-6.15Mn-1.5Si	[75]

Proposed in 2003 by J.G. Speer *et al.* [76], quenching and partitioning (Q&P) steel is a promising material due to its potential in realizing high mechanical properties at relatively low cost. The process of Q&P treatment can be described as following: a fully or partially austenitized steel is quenched to a temperature between martensite start (M_s) and martensite finish (M_f) temperature range, then held isothermally at the same or higher temperature (partitioning step), and finally quenched to room temperature. During the partitioning step, carbon atoms in the martensite formed in the first quenching diffuse into austenite to stabilize it against phase transformation in the last quenching step. After the Q&P treatment, the final microstructure consists of a martensite matrix with embedded RA. Additionally, ferrite is present if the steel was intercritically annealed. The martensite in Q&P steel can be classified into two categories: i.e. tempered martensite (TM, tempered during partitioning process, also called primary martensite) formed in the first quenching process and untempered martensite (UM, also called secondary/fresh martensite) generated during the last Q&P step. Bainite and/or carbides may appear

¹ It should be noted that two types of martensite are present in Q&P steels as described below.

² Two types of ferrite were reported in [72].

depending on the specific Q&P parameters used [77]. The typical microstructure of Q&P steel is shown in Fig. 1.11 [71]. Q&P steels have a kind of composite microstructure, where hard martensite matrix controls the strength and soft RA regulates the ductility. In Q&P steels, carbon is the most effective element to modify the final microstructure and phase fractions. Normally, Q&P steels have chemical compositions based on Fe-C-Mn ternary system, where carbon content is lower than 0.5 wt.% and manganese less than 5 wt.% [69–71,78]. Small content of silicon and/or aluminum (< 3.0 wt%) has to be added as well to retard the formation of carbides during partitioning [79,80]. It should be noted that the Q&P process can be applied to other steel grades which were not initially designed for this method, such as press hardening steel [81], Medium Mn steel [82], martensitic stainless steel [83,84], etc.

Fig. 1.11 (a) SEM image and (b) EBSD phase map of Q&P processed steel (Fe-0.2C-4.0Mn-1.6Si-1.0Cr, wt.%). γ and α'_p refer to RA and primary/fresh martensite, as marked in red and green in (b), respectively [71].

Medium manganese steels are recognized as a promising candidate of the third generation AHSSs due to their attractive properties and affordable cost [85–87]. The ‘medium Mn’ generally means 3~10 wt.% Mn addition [85]. It is believed that R.L. Miller [88] firstly reported the investigation on steels with medium Mn content. The main

1.1 Advanced high strength steels (AHSSs)

microconstituents in medium Mn steels are ferrite and RA after intercritical annealing [73,88]. Martensite [74] and carbides [89] may appear as well depending on the chemical composition and applied processing route. The RA fraction has a wide range in medium Mn steel, 10~40% [88,89], offering flexibility on regulating mechanical properties. For a specific medium Mn steel, there exists an optimum annealing temperature to obtain the maximum RA fraction [88,89]. Hot or cold rolling process also has strong influence on the final microstructure and mechanical properties [73,90]. Fig. 1.12 shows the different microstructure morphology of hot and cold rolled medium Mn steel followed by annealing treatment [73]. The absence of recrystallization in hot-rolled and annealed specimen resulted in lath-shaped grains [73].

Fig. 1.12 EBSD IQ-phase maps of an intercritically annealed Fe-0.093C-7.22Mn-0.49Si (wt.%) steel after (a) hot and (b) cold rolling. Austenite is in green and ferrite in red. IQ: image quality. The blue and black lines are grain boundaries with misorientation angles of 3°~15° and >15°, respectively [73].

Considerable expectations are pinned on the third generation AHSSs to satisfy the requirement of lightweighting for vehicle bodies. Scientists are still paving the way for their applications [56]. One critical point for these steels is the austenite stability, which influences their final strength, ductility and strain hardening ability. More efforts are needed to optimize the alloy design and processing routes for the industrial implementation of these new AHSSs.

1.2 Mechanical properties of AHSSs

The AHSSs have been widely used in vehicles, as they have the capability to meet different requirements for automotive components [3]. Before a steel sheet is shaped into automobile parts, extensive mechanical testing must be completed and the results must be thoroughly analyzed to ensure there will be no unexpected failure during service. Several most important mechanical properties, which are relevant to automotive applications of AHSSs, are reviewed in the following chapters.

1.2.1 Strength and ductility

The AHSSs have yield strength (YS) >300 MPa and ultimate tensile strength (UTS) >600 MPa [4]. Fig. 1.13 summarizes the tensile strength and elongation data of each generation AHSSs extracted from publications. It is seen that the mechanical properties of these steels are overlapping with each other. The generally acknowledged performance of AHSSs is following: the first generation AHSSs have high strength but insufficient ductility, the second generation AHSSs make up the shortcomings in ductility without sacrificing strength, while the third generation have mechanical properties between the first and second generation AHSSs.

Fig. 1.13 Tensile mechanical properties of various AHSSs from existing literature: DP steel [16,19,91-97], TRIP steel [19-21,96-104], CP steel [22-25,97,105-108], MART steel [23,91,96,97,109-112], TWIP steel [37-39,46,47,96,113-116], AUST. SS [117-124], L-IP steel [33,34,41,42,67,125], Q&P steel [69-71,126,127] and Medium Mn steel [75,128-130]. EL_t: total elongation. UTS: ultimate tensile strength.

1.2 Mechanical properties of AHSSs

Among the first generation AHSSs, MART steel has the highest UTS value (up to 1700 MPa), followed by CP steel, then TRIP and DP steels, as shown in Fig. 1.13. The UTS scopes of DP and TRIP steels are similar, lying within the range of 600~1300 MPa (green and blue circles in Fig. 1.13), while for CP steel, the UTS range is 800~1300 MPa. The very high strength of MART steel is provided by martensite, which has a distorted bcc lattice supersaturated with carbon atoms and also confines the EL_t of MART steel to less than 10% (cyan circles in Fig. 1.13). Except TRIP steel, the other three steels (DP, CP and MART steel) have limited strain hardening effect, as dislocation glide is their main deformation mechanism. Therefore, the UTS properties of the three steels are significantly influenced by the hard phase in them, which is usually martensite (as shown in Table 1.1). For example, the linear relation between UTS and martensite fraction in DP steel has been reported widely [13,14]. The EL_t of DP and CP steel has strong dependence on their soft phases, such as ferrite. Grain size is another aspect which affects the ductility of DP, CP, MART as well as other steels, because refined grains hinder dislocation glide and increase ductility and strength at the same time. For TRIP steel, both the elongation and strength are enhanced by martensitic transformation process of metastable austenite (TRIP effect) during plastic deformation. The TRIP phenomenon takes effect from: 1) the shear strain and volume change on microscale accommodate macro deformation, and thus postpone the onset of necking; 2) the newly formed martensite after phase transformation has higher hardness than austenite, resulting in considerable improvement in strain hardening. The former improves the ductility while the latter benefits the mechanical strength of TRIP steel.

In the second generation AHSSs, AUST. SS has the lowest UTS (600~800 MPa), while L-IP and TWIP steel has wider scopes in UTS (600~1300), as shown in Fig. 1.13. On the other hand, these three steels possess excellent ductility under tensile loading, and most reported EL_t values are in the range of 40%~70% (Fig. 1.13). The excellent mechanical performance of these steels stems from the deformation of austenite. All three members

of the second generation AHSS are fully or partially composed of austenite (seen in Table 1.2), therefore their mechanical properties are closely related with the deformation behavior of austenite. Depending on SFE, austenite grains deform through martensitic phase transformation, twinning and dislocation glide with increasing SFE value [44]. The SFE of metals is well-known to be determined by chemical composition and temperature [131]. The difference in SFE between TWIP and AUST. SS leads to that the mechanical twinning (TWIP effect) is active in TWIP steels, while both phase transformation (TRIP effect) and TWIP effect are operating during plastic deformation of AUST. SSs. Deformation twinning effect increases the strength of steels through impeding dislocation glide, since twin boundaries are insurmountable barriers for dislocation movement because of their essence of high angle boundaries (with misorientation $>15^\circ$). The formation of mechanical twins under deformation leads to: 1) extensive global plastic deformation via shear strain inside austenite grains; 2) the accommodation of external loading, as shear stress is needed for twinning process; 3) considerable grain refinement by highly dense twins. The thickness of deformation twins is down to tens of nanometers in TWIP steel [132], significantly decreasing the maximum distance for dislocation glide. The deformation twins with high density and their strong effect on dislocation glide lead to the so-called “dynamic Hall-Petch effect” [44]. With the assistance of mechanical twins, attractive properties with high UTS (800~1000 MPa) and EL_t (50%~70%) were readily achieved in TWIP steels.

Depending on the actual SFE and added alloying elements, TRIP and/or TWIP phenomena develop and provide strain hardening during deformation in L-IP steel as well [41,67]. However, in L-IP steel with high SFE ($>90 \text{ mJ/m}^2$), shear band induced plasticity (SBIP) mechanism dominates the strain hardening process [133]. TEM investigations show that the shear bands (also called microbands) are thin planar shear zones which are confined by dislocation walls on either side [133,134]. These microbands handicap dislocation glide and refine grains, improving both strength and ductility of the involved

steels.

Q&P steel has outstanding strength (900~1700 MPa), which is comparable to MART steel, and also good ductility (10%~25%) which MART steel does not have. Medium Mn steel has lower strength (800~1400 MPa) than the Q&P steel, but its ductility is higher (10%~40%). The main phases in Q&P steel are hard martensite and soft RA, where the former controls the strength and the latter regulates ductility. In Medium Mn steel, the main hard phase is ferrite and the soft one is the same as in Q&P steel, i.e., RA. In these two steels, RA transforms into martensite under loading, strengthening the steel and offering plasticity, namely TRIP effect. The mechanical stability of RA, i.e., the resistance to mechanically induced martensitic transformation, is dependent on its chemical composition [135], size [136], shape and spatial location in the microstructure [127]. The strength and ductility of Q&P steel change with the variation of stability and volume fraction of RA [70,126]. Similar modification on mechanical properties of Medium Mn steel was reported as well through adjusting the phase ratio and RA stability [137]. Additionally, the investigation on grain refinement via controlling rolling and annealing process have attracted much attention [137,138], as ultrafine grains increase the strength and ductility simultaneously.

1.2.2 Formability of AHSSs

Thin sheet is the most widely used form of steel in automotive industry. Several basic sheet forming methods, such as blanking [139], flanging [140], bending [141] and deep drawing [142], are frequently employed to produce vehicle components from AHSS sheets. Blank steel sheets are formed into complex geometry with desired tolerances and properties after undergoing deformation under multiple stress states involving tension, compression, shear, etc. Steel sheets must possess the capability to withstand the complex straining modes without cracking and failure during forming process, namely the formability. Therefore, it is of great importance to investigate the formability of

AHSSs before their applications. A number of methods for evaluation of formability were proposed and partially standardized, including forming limit curve/diagram (FLC/D) [143,144], drawability [145] and stretch flangeability [146]. Among these, FLC is the well-known and most widely used one describing the formability of automobile steel sheets. FLC represents the critical strains that sheet material can withstand before failure during in-plane deformation [147]. The FLC covers failure limits for different principal strain ratios/strain paths (Fig. 1.14), such as equi-biaxial tension ($\epsilon_1 = \epsilon_2$), plane strain ($\epsilon_2 = 0$), uniaxial strain ($\epsilon_1 = -2\epsilon_2$) and pure shear ($\epsilon_1 = -\epsilon_2$), which are commonly observed during steel sheet forming process [147]. The FLC is usually obtained from static testing, such as Nakazima test [148] and Marciniak test [149]. The main difference between the two approaches is that the former uses a hemispherical punch while the latter uses flat-faced cylindrical punch [147]. The FLC of various AHSSs obtained by Nakazima test are shortly overviewed below.

Fig. 1.14 Typical forming limit curve and grid analysis used to calculate strains during Nakazima or Marciniak testing [147].

A large number of investigations on FLC of DP and TRIP steel have been carried out [150–152]. A. Konieczny [150] comparatively studied the formability of two hot-dipped DP steels (DP 780 and DP 580) with thickness of 1.2 mm and showed that DP 590 has better formability in all strain paths than DP 780, as shown in Fig. 1.15. However, no detailed explanation for such difference was provided. D. Schwindt *et al.* [151] obtained

1.2 Mechanical properties of AHSSs

FLC of a 1.1 mm thick DP 780 steel, and they found that the reduced sample shape used in the experiments resulted in an overestimation on the FLC of the DP 780 steel when the thickness was more than 1 mm. C.G. Lee *et al.* [152] investigated the influence of volume fraction and stability of RA on the formability of a 0.8 mm thick TRIP steel with UTS of ~ 720 MPa and EL_t of $\sim 30\%$. They found that the relationship between the formability and the fraction and stability of RA is plausible. The increasing RA volume fraction improved the formability of the steel. Better formability was achieved in specimens with higher RA stability when the RA volume fraction was the same. They suggested that attention should be paid to RA stability when designing the microstructure of TRIP steel.

Fig. 1.15 Forming limit curves of (a) DP 590 and (b) DP 780 steel obtained from Nakazima test [150].

Tremendous research works demonstrated that the second generation AHSSs have superior formability, which is desired for automotive industry. N. Habibi *et al.* [153] studied the formability of a Fe-15Mn-2Al-0.6C (wt. %) TWIP steel with thickness of 1.4 mm by Nakazima testing as well as by simulation employing theoretical models. The FLC obtained from experiments is presented in Fig. 1.16, where the minimum strain to failure is about ~ 0.42 , exhibiting excellent formability. They concluded that: 1) the models which only consider ductile or shear fracture criterion are unable to properly predict the

fracture FLC; 2) other aspects, including the maximum shear stress, void nucleation, growth and coalescence during steel sheet forming, must be taken into account to calculate FLC using ductile fracture criteria. K. Chung *et al.* [154] experimentally obtained the FLCs of TWIP and DP steel, and the results showed that the maximum plane strain (when minor strain is zero) of TWIP 940 is about 0.38 while for DP 600 the value is 0.25, indicating much better formability of TWIP steel. M. R. Rocha *et al.* [155] carried out Nakazima test on two austenitic 304 and 304 N stainless steels and also studied the influence of strain path on martensitic transformation via magnetic and XRD techniques. Their tests revealed: 1) both AUST. SSs have excellent formability with maximum plane strain ~ 0.55 ; 2) tension-tension loading favors the formation of α' -martensite compared to the tension-compression; 3) a larger quantity of ε -martensite was detected by XRD in compressive deformation than that in tensile loading.

Fig. 1.16 Forming limit curve of a TWIP steel [153].

In the last decade, the formability of the third generation AHSSs has drawn much attention [156–160]. It is generally believed that the third generation AHSSs have satisfactory formability. B. Mohammed *et al.* [156] investigated the formability of a Fe-0.2C-1.8Mn-1.5Si (wt.%) Q&P steel by combining crystal plasticity finite element model and digital image correlation (DIC) equipped Nakazima test. It was found that: 1) the maximum plane strain in FLC is 0.25 even though the UTS of the studied Q&P is as high

1.2 Mechanical properties of AHSSs

as 1019 MPa, exhibiting excellent formability, as shown in Fig. 1.17; 2) the developed model, which was microstructure-sensitive, accurately predicted the strain at the onset of necking, providing a promising tool for FLC calculation. The FLC of Q&P 980 steel obtained by L. Zhang *et al.* [157] indicated the maximum plane strain of 0.21, which is similar to that of DP 980 steel. Another research work on Q&P 980 steel done by R. Liu *et al.* [158] focused on the effect of punch speed on FLC. They found that higher formability is achieved in Q&P steel at lower speed of 1 mm/s than that at 120 mm/s. They also recommended that the formability determination should be performed at strain rates of $1\sim 10\text{ s}^{-1}$, which are relevant to the industrial stamping process. X.L Gao *et al.* [159] employed several criteria to predict the FLC of Q&P 980 steel, and the results indicated that the maximum tensile stress criterion (MTSC) shows the best agreement with the experimental results. G. Zheng *et al.* [160] investigated the influence of testing temperature in the range of 300~500 °C on the formability of a Fe-0.1C-5.0Mn-0.03Al (wt.%) steel. The optimum formability of the studied medium Mn steel was obtained at 400 °C.

Fig. 1.17 Forming limit curve of a Q&P 980 steel. Specimens were machined in three directions: 0°, 45°, and 90° with respect to rolling direction [156].

It can be outlined that the second generation AHSSs have the highest formability among three generations of AHSS, which is mainly derived from the deformation capability of austenite. Necking and cracking are retarded to a larger extent in second generation AHSS compared with other generations. The formability of the first generation AHSSs is different for each steel. For instance, TRIP steel has higher forming limit than

CP and MART, good formability can be obtained in DP steel at the expense of strength. Additionally, the third generation AHSSs tend to show satisfactory formability for automotive industry and scientists are endeavoring to realize good formability with affordable cost.

1.2.3 Fatigue of AHSSs

Fatigue refers to the failure or changes in properties that occurs in materials subjected to fluctuating or cyclic stresses [161]. Fatigue problem caught the attention of engineers as early as ~200 years ago when the first recorded publication on fatigue test was finished by Albert in 1837 [162,163]. Nowadays, tremendous testing methods and theories have been proposed on fatigue of materials. In order to imitate the service condition of structures or components, various cyclic loading were applied to specimens by fatigue testing machines, such as axial tension-compression, bending or torsion [164]. The fatigue testing results are generally depicted as S-N curves (also called stress-life curves), namely stress amplitude versus logarithm of the number of cycles to fatigue failure, as schematically shown in Fig. 1.18. Stress amplitude (σ_a) refers to one half of the range between the maximum (σ_{max}) and minimum (σ_{min}) stress in cycles, i.e., $\sigma_a = (\sigma_{max} - \sigma_{min})/2$. The fatigue limit (S_f) is the stress amplitude (i.e. the plateau of the S-N curve) at/below which the sample may be cycled indefinitely without failure. In practice, many alloys do not have a plateau on their S-N curves. In these cases, the corresponding stress level at which failure will not occur for a specified number of cycles (e.g., 10^6 or 10^7 cycles) is defined as the fatigue strength of the material. Furthermore, fatigue is usually divided into two regimes, i.e., high-cycle fatigue (HCF) and low-cycle fatigue (LCF). HCF is associated with fatigue lives greater than $10^4 \sim 10^5$ cycles while that in LCF is lower than this range [165]. The process of fatigue failure is characterized by four stages: (1) cyclic hardening or softening; (2) crack initiation; (3) crack propagation and (4) final failure. The rough regimes for these processes on S-N curves are presented in Fig. 1.18. The fatigue performance of AHSSs is of great importance as cyclic loading is one common

loading approach in the everyday service of vehicle components. In this chapter, the fatigue performance of various AHSSs is briefly introduced.

Fig. 1.18 Typical S-N curve schematically showing the four stages of fatigue in ductile metals until failure [166].

The study on DP steel done by K. V. Sudhakar *et al.* [167] showed that the fatigue crack growth rate decreased with increasing martensite volume fraction in DP steel. They concluded that the low carbon content in martensite, formed during quenching after intercritical annealing, resulted in retarding of crack growth by crack tip blunting and/or deflection. A. M. Sherman *et al.* [168] investigated the influence of pre-strain on the fatigue properties of a vanadium added DP steel. They found that the pre-strain of 8% has little effect on the cyclic stress-strain curve, S-N curve and notch sensitivity, while monotonic increase of tensile strength was observed after pre-straining. The effect of microstructure on LCF of a Fe-0.10C-1.26Mn-0.66Si (wt.%) DP steel was investigated by S. R. Mediratta *et al.* [169]. They revealed that the microstructure with fine dispersion of martensite in fine-grained ferrite resulted in the best fatigue resistance in strain controlled fatigue testing. G.N. Haidemenopoulos *et al.* [170] studied the effects of RA volume fraction and stability on fatigue performance of TRIP 700 grade. The obtained S-N curve is presented in Fig. 1.19. The experiments revealed that: 1) martensitic transformation (TRIP effect) took place during HCF; 2) higher fatigue performance was achieved with higher RA stability even when the RA fraction was lower. The influence of pre-strain on the fatigue of TRIP 780 steel was carried out by L.T. Robertson *et al.* [171].

They drew the following conclusions: 1) the TRIP effect during pre-straining enhanced the cyclic strength which, in turn, reduced the softening effect during LCF; 2) pre-strain has improved fatigue life of TRIP steel under high strain amplitudes but has had little effect at lower strain amplitudes.

Fig. 1.19 S-N curves of TRIP 700 steels with different treatments. The volume fraction of retained austenite after fatigue testing is shown in parentheses [170]. Treatment A and B are 420 s holding at 400 °C and 120 s holding at 460 °C, respectively.

TWIP steels demonstrate better fatigue performance, as shown in Fig. 1.20a reported by Y.W. Kim *et al.* [172]. Their experiments demonstrated that both the LCF and HCF resistance were strongly dependent on pre-strain but exhibited different effects: LCF resistance was degraded but HCF resistance was improved with increasing pre-strain. The reason was attributed to the increase in strength and decrease in ductility after pre-straining. In another study on TWIP steel done by T. Niendorf *et al.* [173], the enhanced fatigue performance after pre-straining is ascribed to the increased interaction of glide dislocations with twins of increased density due to the pre-deformation. While in no pre-strained TWIP steel, dislocation movement gives way to the growth of the existing twins and no new twin nucleates, leading to softening under cyclic loading [173]. The grain size effect on the fatigue property of 304 stainless steel (SS) was investigated by A. Di Schino *et al.* [174] and the experimental results revealed that the fatigue resistance was improved evidently with decreasing grain size, as shown in Fig. 1.20b. K.B. Sankara Rao *et al.* [175] examined the effect of pre-strain on the LCF of 304 SS. The steel was cold worked to three

1.2 Mechanical properties of AHSSs

level, i.e. 10%, 20% and 30%, and then tested under strain controlled mode in the temperature range of 27~750 °C. The experiments indicated: 1) the strong independence of fatigue life of 304 SS on cold working at the study temperature range; 2) continuous cyclic softening until failure on the pre-strained material at 27 and 550 °C; 3) the crack initiation and propagation modes at 27 and 550 °C is transgranular.

Fig. 1.20 (a) The effect of pre-strain on the HCF life behavior of TWIP steel. Arrows indicate that the test specimens did not fail [172]. (b) Grain size dependence of the fatigue resistance of AISI 304 stainless steel [174].

I. de Diego-Calderón *et al.* [176] thoroughly investigated the effect of RA on the HCF performance of two Q&P steels and their S-N curves are shown in Fig. 1.21. The experiments demonstrated that: 1) the fatigue limit of the steel was benefited by the increased RA fraction due to the delay effect of martensitic transformation on crack propagation; 2) the austenite-martensite transformation was promoted with increasing stress amplitude during cyclic loading; 3) the RA stability under cyclic stress is mainly determined by its size and crystallographic orientation while scarcely by its shape and distribution; 4) intergranular and transgranular mechanisms control fatigue crack formation under low and higher stress amplitude, respectively; 5) fatigue crack propagates transgranularly at all stress amplitudes. P. Matteis *et al.* [177] comparatively studied the fatigue properties of Q&P, TWIP and DP steels with similar ultimate tensile strength but different ductility. The S-N curves revealed that Q&P and DP possessed similar fatigue strength (570 MPa) which was much higher than that of TWIP steel (410 MPa). The excellent fatigue performance of Q&P steel is also illustrated on Fig. 1.22, where the measured fatigue strength of newly designed Q&P steels are in the range of 700~900

MPa with fatigue ratio more than 0.6. The experimental study on a medium Mn steel by X.Y. Qi *et al.* [178] revealed that: 1) the secondary cracks ahead of the main fatigue crack reduced the growth rate of the main crack by decreasing stress concentration, resulting in the enhanced fatigue strength of the steel; 2) the increasing stress amplitude increased the martensitic transformation rate of RA due to the rising driving force for RA transformation; 3) RA improved the fatigue strength of the medium Mn steel by relaxing local stress, blunting crack tip, delaying crack initiation and decreasing crack propagation rate.

Fig. 1.21 S-N curves of Q&P-0.25C and Q&P-0.28C steel grades in comparison [176].

Fig. 1.22 The relationship between fatigue strength and UTS for different AHSSs. The red dashed lines are the ratios of fatigue strength and UTS from 0.4 to 0.9, and two of them are marked as examples.

The fatigue limits of AHSSs are almost linearly proportional to their strength, as demonstrated in Fig. 1.22. Of course, the influences of other aspects such as grain size, surface roughness or internal microcracks, metallurgical quality etc., should not be

1.2 Mechanical properties of AHSSs

ignored. The relatively low YS of the second generation AHSSs put them in a disadvantaged position where higher fatigue strength is needed. As mentioned before, their fatigue performance can be improved via work hardening. For the first and third generation AHSSs, their fatigue properties are comparable. Furthermore, the dispersion and irregularity in test data make it hard to compare fatigue performance in detail between different generations of AHSSs.

1.2.4 Fracture toughness of AHSSs

After multiple heat treatments and metalforming operations, micro- or macro-cracks may develop in steel components internally or peripherally due to residual stress concentrations or inclusions. For example, one of the frequent cracking sources is welding which is widely used in automotive industry. Cracks are hardly or uneconomically to be completely removed in AHSSs and they may coexist with automobile components loaded under various stress states during service life. Therefore, knowing more about fracture toughness and resistance of AHSSs is critical for their automotive application. However, automobile steel sheets normally do not fulfill the requirements of ASTM E-399 toughness testing standard [179] as they usually have a thickness less than 5 mm after final rolling. Different approaches have been proposed to investigate the fracture toughness and resistance for AHSS sheets [167,180,181]. In this section, the relevant studies on fracture resistance of AHSSs are briefly overviewed.

The strong dependence of fracture toughness of DP steels on martensite volume fraction has been reported by several authors. A. Bag *et al.* [180] investigated the influence of martensite fraction on static and dynamic fracture toughness of a high martensite content DP steel using chevron-notched specimens. The obtained toughness values are plotted vs. martensite fraction in Fig. 1.23. The experiments revealed that the static fracture toughness reached the peak at martensite volume fraction $V_m=60\%$ in the studied range $V_m=30\%\sim 80\%$, while at dynamic state, the fracture toughness increased as the steel contains martensite $V_m=45\%\sim 60\%$ and then achieved a saturation plateau. Another study

was reported by K. V. Sudhakar *et al.* [167] using half-sized specimens. They concluded that the DP steel with martensite volume fraction of 76% shows the best combination of strength and fracture toughness. Y.J. Chao *et al.* [159] calculated the fracture toughness (K_{IC}) of DP 590 steel from corrected sub-sized Charpy impact testing, and the results showed that the K_{IC} value of DP 590 steel at room temperature is approximately in the range of 160~200 $\text{MPa} \cdot \text{m}^{1/2}$, indicating excellent toughness.

Fig. 1.23 The variation of (a) static (b) dynamic fracture-toughness vs. volume fraction of martensite in DP steel. K_{ID} : plain strain fracture toughness; K_D : equivalent energy fracture-toughness; K_{ID} : J-integral dynamic fracture-toughness; K_{ICV} : Chevron-notched fracture toughness. K_{ID} , K_D are K_{ID} are dynamic fracture toughness calculated from load-time and energy-time curves of Charpy impact testing, while K_{ICV} is static one obtained from three-point bend testing using Chevron-notched specimens [180].

In AUST. SSs, a few frequently used processes, such as cold working and aging, may lead to unexpected deterioration of fracture toughness. The negative effect of cold working on fracture toughness was observed in 304 and 316 stainless steel even though the strength of two steels was evidently improved [182]. The origin of this adverse effect is that inclusion alignment was exaggerated during cold deformation [182]. Aging treatment at 550 °C and 566 °C resulted in 20%~35% degradation in toughness (J_c) of AUST. SSs compared to unaged samples because of the cracks initiated at grain boundaries containing $M_{23}C_6$ carbides formed during aging treatment. The experimental study of the elastic-plastic fracture toughness (J_c) of 304 SS and 316 SS by W.J. Mills [183] revealed that both materials have high fracture resistance and the author suggested that the fracture control is not a primary design consideration for AUST. SSs. Furthermore, the high variability was also reported in J-integral of 304 SS via finite element method

(FEM) simulation [184] and experiments [183], however, the reasons are needed to be elucidated.

Except the enhancement of strength and ductility, Q&P treatment improves fracture toughness as well. The K_{IC} value of a 1200 MPa Q&P steel was measured experimentally by using double edge-notched tension (DENT) specimens [185]. And obvious enhancement was found on the steel as the K_{IC} of the Q&P processed steel is $74.1 \text{ MPa} \cdot \text{m}^{1/2}$, which is by 32% and 16% higher than the K_{IC} of QT (quenching and tempering) and QAT (quenching and austempering) treated steels, respectively [185]. The crack propagation process under loading in Q&P steel is schematically shown in Fig. 1.24. Whether the RA around crack tip area transforms is dependent on its stability and the low energy routes for propagation is preferential for cracks [185]. However, contrary to the favorable effect on strength and ductility, RA in Q&P steel may have negative effect on fracture toughness. Martensitic transformation (TRIP effect) of less stable RA occurred preferentially at the tips of cracks under tension loading, and this process further enhanced the toughness of Q&P steel, but the phase transformation of more stable RA has detrimental effect on toughness [185]. The similar deleterious effect of RA stability on fracture resistance was also confirmed by P. Jacques *et al.* [181]. The authors obtained lower fracture toughness in the steel with higher strength-ductility balance [181]. The decreased fracture toughness was attributed to the nearly continuous network containing brittle martensite developed in the fracture process zone, where faster crack initiation was induced by premature cracking of martensite [181]. Therefore, the authors suggested that, to achieve the fracture resistance of steels involving RA and TRIP effect, the amount of RA should be limited to avoid premature damaging from the “brittle network” effect [181].

Fig. 1.24 Illustrations of voids nucleation and coalesce process taking place in the plastic zone during DENT tests. (a) Initial state before loading—there is no evident deformation; (b) after loading and before void nucleation, the crack is blunted; (c) as the load increases, the voids nucleate dispersedly; (d) finally, the voids coalesce and crack propagates [185].

It can be outlined that the first generation AHSSs have insufficient toughness due to high fraction of ‘hard/brittle’ martensite. The soft austenitic matrix of the second generation AHSSs results in their very high fracture toughness. More studies are needed to be carried out on the fracture toughness of Q&P steel or RA-assisted AHSSs, as the effect of RA on crack propagation is still a matter of debate [181,186]. Martensitic transformation of RA absorbs energy postponing the crack propagation process but the newly formed martensite is brittle which may accelerate the crack process. This process could be detrimental for the toughness of the third generation AHSSs, as they rely heavily on RA to provide ductility and toughness.

1.3 High strain rate performance and impact resistance of AHSSs

High speed deformation is unavoidable for AHSSs during manufacturing processes, such as stamping, and crash accidents. Mechanical properties and deformation behavior of materials under high strain rate are usually different from those at static conditions. For instance, metals with body centered cubic (bcc) structure show high strain rate sensitivity, namely flow stress increases with rising strain rate at the same strain, while metals with face centered cubic (fcc) structure are not sensitive to strain rate variation. Furthermore, bcc and fcc phases may occur simultaneously in one AHSS grade, such as martensite and austenite in Q&P steel, leading to complex mechanical response at high

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strain rates. Therefore, it is of great significance to shed light on the mechanical properties and deformation behavior of AHSSs at high strain rates. The response of AHSSs under dynamic loading is overviewed below.

1.3.1 High strain rate tensile behavior

Large amount of investigations on dynamic tensile behavior of AHSSs were reported by employing split Hopkinson tensile bar (SHTB) testing apparatus. Experimental investigation on 600/800/1000 MPa DP steels and a 1200 MPa fully MART steel demonstrated the positive strain rate sensitivity in DP steels (as shown in Fig. 1.25), while negative strain rate sensitivity was observed for martensitic steels [187]. The positive influence of strain rate on flow stress was also verified in DP 600 and DP 800 steels in [188,189]. The microstructure characterization showed that, in comparison to the microstructure deformed at low strain rates, the ferrite grain elongation decreased and might result in lower ductility at high strain rates in DP 600 and DP 800 steels [189]. In another study, a significant strain rate sensitivity was reported for a tempered martensitic steel in [190]. E. Cadoni *et al.* [190] also simulated the high strain rate deformation of a MART steel using a model based on net dislocations but without taking into account the adiabatic heating effect. This could be the origin of the inconsistent estimation of strain rate sensitivity as the elevated temperature does affect flow stress. Study of four low carbon TRIP steels [20] and three different TRIP steels in [191] showed that their mechanical properties increase with increasing strain rate. Furthermore, the enhancement was revealed on ferrite grain elongation and martensitic transformation of RA in TRIP 600 and TRIP 800 steel at higher strain rates than at static condition [189], implying better ductility as well.

Fig. 1.25 The engineering stress-strain curves of (a) DP 600 and (b) DP 800 steel under dynamic and static tensile tests [187].

The mechanical response of Fe-30Mn-3Si-4Al (wt.%) TWIP steel at strain rates of $700\sim 5000\text{ s}^{-1}$ was investigated by Z.P. Xiong *et al.* [192]. They reported that: 1) The strain hardening effect dominates the process when strain rate is in the range of $700\sim 2500\text{ s}^{-1}$ while strain rate softening developed after further increasing the strain rate to 5000 s^{-1} ; 2) the density of deformation twins increased to its maximum at 2500 s^{-1} and slightly decreased when the strain rate changed from 700 s^{-1} to 5000 s^{-1} ; 3) the temperature increase due to adiabatic heating saturated at strain rate of 2500 s^{-1} . J. Park *et al.* [38] studied the mechanical response and microstructure evolution of TWIP steel at strain rates of $10^{-3}\sim 2000\text{ s}^{-1}$ using interrupted tensile tests. Positive strain rate sensitivity was shown by the studied TWIP steel. EBSD examination revealed that mechanical twins formed at 20% strain under static tension in TWIP steel, while twins formed earlier at 10% strain during dynamic tensile deformation [38]. The sensitivity of flow stress to strain rate in 304L SS is dependent on plastic strain, as shown in Fig. 1.26 by J.A. Lichtenfeld [193]. The experiments revealed that positive strain rate sensitivity was observed when the true strain $< 25\%$, which was attributed to the competition between hardening from TRIP effect and softening from adiabatic heating [193]. Experimental investigation on AUST. 304 SS at strain rates of $10^{-3}\sim 10^2\text{ s}^{-1}$ revealed that the maximum martensite volume fraction measured in necking zone is $\sim 50\%$ and $\sim 30\%$ after static and dynamic deformation, respectively [194]. It is reasonable to presume that the transformation from austenite to martensite (TRIP) was suppressed and dislocation activity dominated the deformation of 304 SS under high strain rate tensile loading [194]. Furthermore, the

1.3 High strain rate performance and impact resistance of AHSSs

detailed study done by K. P. Staudhammer *et al.* [195] demonstrated that more martensite was transformed at high strain rates than that at static loading when strain was $<25\%$, but the case reversed when strain was $>25\%$.

Fig. 1.26 True stress vs. true strain curves from tensile testing of 304L steel at various strain rates [193].

The investigation on Q&P 980 steel indicated that the flow stress of steel exhibited evident strain rate sensitivity (as shown in Fig. 1.27a) while martensitic transformation of RA showed non-monotonic effect with strain rate variation [196]. XRD characterization revealed that, with increasing strain rate in the range of $2 \times 10^{-4} \sim 10^{-1} \text{ s}^{-1}$, the RA transformation rate slowed down due to heating effect, but the acceleration of RA transformation was observed in strain rate range of $10^{-1} \sim 1.75 \times 10^2 \text{ s}^{-1}$ because of more martensite nucleation sites [196], as presented in Fig. 1.27b. The suppression of TRIP effect due to increased strain rate was also reported in [197], where the RA transformation was retarded when the strain rate reached to 500 s^{-1} . However, such effect of strain rate on RA transformation was not observed in Q&P 980 steel studied by interrupted SHTB tests [198]. It was shown that RA fraction reduced exponentially with increasing strain under both static and dynamic tensile loading, but no strain rate sensitivity was noticed [198]. The strain rate effect on elongation of Q&P steel is more complex. The experiments done by C. Liu *et al.* [199] indicated that the total elongation decreased with increasing strain rate in the range of $10^{-4} \sim 10^{-1} \text{ s}^{-1}$, raised to the maximum

at $8 \times 10^1 \text{ s}^{-1}$, and decreased again in the range of $10^2 \sim 10^3 \text{ s}^{-1}$. Such intricate relation was attributed to the influence of strain rate on TRIP process in Q&P steel [199].

Fig. 1.27 (a) Engineering stress-strain curves for Q&P steel in tension at various strain rates. (b) RA fraction at different strain under static and dynamic tensile testing for Q&P steel [196].

It can be outlined that almost all AHSSs exhibit positive strain rate sensitivity, i.e. the flow stress increases with increasing strain rates, though the sensitivity index differs from steel to steel. This is advantageous for automotive applications as higher energy will be absorbed by AHSSs components in accidents compared with that at static condition. However, due to the oscillation problem at high speed testing, accurate measurements of flow stress and strain rate sensitivity maybe difficult. Additionally, special attention should be paid to the variation in deformation mechanisms from static to dynamic loading, e.g., the influence of strain rate on TRIP effect in steels containing RA.

1.3.2 Impact resistance

The safety performance of passenger cars is dependent to large extent on the materials used in vehicle bodies. During crash accidents, automobiles must be able to absorb the impact energy as much as possible and keep passenger safe. The materials used in vehicles including AHSSs should withstand certain amount of deformation without failure to consume the energy. Therefore, the impact resistance and deformation behavior under impact loading of AHSSs are of great significance for automotive applications. In this Section, the relevant investigations focused on the impact

1.3 High strain rate performance and impact resistance of AHSSs

performance of AHSSs are overviewed.

For DP steels, their impact resistance has been studied mainly by means of Charpy impact testing [180,200–210], which is a standardized method to evaluate material impact toughness. An overview of Charpy impact energy values at room temperature reported for different DP steels is presented in Table 1.4. As can be seen, the measured values vary dramatically in the range of 1.7–235 J. It was shown that Charpy impact toughness of vanadium-strengthened DP steel was improved via replacement of pearlite by martensite [201]. A. Bag *et al.* found that the Charpy V-notched impact energy of a Fe-0.16C-1.32Mn-0.44Si (wt.%) DP steel is maximum at 60% martensite volume fraction [200]. Simulations conducted by Y. Prawoto *et al.* using Johnson-Cook model led to the similar optimum martensite volume fraction for enhanced Charpy impact resistance [202].

Table 1.4 Values of Charpy impact energy measured on various DP steels. (S: sample section size; T: test temperature; CVN: Charpy V-notched impact energy)

Chemical composition (wt.%)	S (mm)	T (°C)	CVN (J)	Ref.
Fe-0.16C-1.32Mn-0.44Si-0.09Mo	10×10	25	96	[200]
Fe-0.14C-1.23Mn-0.34Si-0.10V	6.25×10	25	189	[201]
Fe-0.08C-1.24Mn-0.09Si-1.23Cr	5.5×10	20	70	[204]
Fe-0.15C-0.90Mn-0.25Si-0.10Mo-0.80Cr-1.20Ni	10×10	25	109	[207]
Fe-0.08C-1.24Mn-1.14Si-0.87Cr	5.5×10	31	70	[210]
Fe-0.16C-1.23Mn	10×10	25	150	[211]
Fe-0.17C-1.49Mn-0.22Si	3×4	25	1.7	[212]
Fe-0.07C-1.52Mn-0.34Si-0.05Nb-0.05V	10×10	25	235	[213]

Note: Information on the microstructure (phase volume fraction, grain size, morphology, etc.) is not provided in the table.

Computational modelling confirmed that both the constituent fraction and its morphology have a great influence on the Charpy impact resistance of martensite-ferrite DP steel [202]. Detailed investigations on DP 590 steel revealed that the ductile-brittle transition temperature (DBTT) is $-95\text{ }^{\circ}\text{C}$, which is far below general automobile service temperature [204]. Another study on a hot-rolled DP 590 steel grade concluded that: 1) the Charpy impact energy in ductile-brittle transition range was increased by splitting

caused by silicate and carbide inclusions; 2) splitting reduces DBTT as well [209,210]. The survey on AISI 4340 martensitic steel with different microstructures, generated via adjusting heat treatment parameters, demonstrated that the combination of bainite and ferrite leads to a better Charpy impact performance and tensile ductility than martensite-ferrite or full bainite microstructure [206]. The study on AISI 3115 ferritic steel pointed out that the impact strength of this steel decreases with the increasing intercritical annealing temperature, which affects the microstructure significantly [207]. It should be noted that, bars with dimensions of 10×10×55 mm are used for Charpy impact testing [214,215], so this method cannot be utilized for characterization of common AHSS sheets. Moreover, Charpy test is performed on notched samples, so deformation process is not relevant to the scenario where automobile components are confronted during crash accidents.

There are also studies focused on impact performance of thin-walled [216–220] and flat sheet [221,222] specimens. Thin-walled columns composed of various cross-section shapes with different size were examined due to their similarity with automobile mid-rails. Experimental crush testing of hydroformed thin-wall steel tubes combined with simulation exhibited that DP steels had higher energy absorption capability compared to the high strength low alloy steel (HSLA) 350 and deep drawing quality steel [218]. Modeling and experiments on DP 800 steel manifested that the axial crush performance of thin-walled specimen has great relationship with the shape of the sample (top-hat or square section) [217]. The study on DP steels with tensile strength from 270 MPa to 1470 MPa showed that yield strength is the most influential factor on the bending moment in bending crash testing [223]. J.R. Fekete *et al.* estimated that the energy absorption capability of thin-walled samples increases by 10% when DP 340 is substituted by HSLA 340 steel [219].

Just a few reports on drop weight impact resistance of flat-sheet specimens can be found in literature. J.K. Holmen *et al.* [221] studied the low velocity impact deformation

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process of monolithic and multi-layered DP 600 steel plates. Digital image correlation (DIC) technique revealed that the plates' resistance against perforation is closely connected with the shape of the impactor (e.g. blunt-ended or ogival-ended) [221]. Another successive study completed by that team on single DP 600 sheet demonstrated the significant effect of strain rate and steel grade on the strain distribution during impact deformation [222].

Various investigations indicated that AUST. SSs have exceptional impact resistance at both ambient and cryogenic temperatures [40,224]. The values of impact strength of 304 SS obtained from Charpy test are listed in Table 1.5. It should be noted that the values from different studies are not comparable even with same sample size and testing temperature, as difference does exist in experiment details and microstructure, which all affect the final measured value. It is widely demonstrated by Charpy test that there is no evident decrease in impact strength of AUST. SSs with decreasing temperature, namely the DBTT phenomenon is absent [224,225]. This outstanding feature makes AUST. SSs more favorable for automotive applications than other ferrite or martensite-based steels, which have inadequate impact resistance at low temperature and obvious DBTT effect [40,226]. The possible reason is that fcc austenite has multiple slip systems which do not need thermal activation so that the decreased temperature has little effect on absorbing impact energy [40]. Furthermore, the insensitivity of impact toughness to temperature is more pronounced in TWIP steel than in AUST. SS. For example, Charpy test done in the temperature range of $-196\text{ }^{\circ}\text{C} \sim +200\text{ }^{\circ}\text{C}$ indicated almost no change in impact strength of Fe-25Mn-3Si-3Al (wt.%) TWIP [43], as presented in Fig. 1.28 .

Fig. 1.28 Charpy impact resistance of Fe-25Mn-3Si-3Al (wt.%) TWIP steel as a function of testing temperature [43].

Table 1.5 Charpy impact resistance of AUST. 304 SSs reported in literature. (S: sample section size; T: test temperature; CVN: Charpy V-notched impact energy)

Chemical composition (wt.%)	S (mm)	T (°C)	CVN (J)	Ref.
Fe-0.045C-1.11Mn-18.13Cr-8.25Ni-0.44Si-0.053N	10×10	-20	322	[224]
		25	350	[224]
Fe-0.047C-1.31Mn-17.87Cr-8.29Ni-0.34Si	10×10	35	150	[227]
Fe-0.043C-1.80Mn-18.06Cr-8.06Ni-0.36Si	10×3	25	50	[228]
Fe-0.022C-1.62Mn-18.8Cr-8.3Ni-0.53Si	10×10	-50	223	[225]
		-25	249	[225]
		0	252	[225]
		25	278	[225]

Note: Information on the microstructure (phase volume fraction, grain size, morphology, etc.) is not provided in the table.

Limited reports on the impact strength of Q&P steel can be retrieved and the measured impact resistance values are listed in Table 1.6. Compared with directly quenched steel, the Q&P processed steel exhibited more excellent performance under impact loading in [229]. Two examples are shown in [229]: 1) the impact energy at 23 °C for the Q&P processed steel (Fe-0.2C-2.0Mn-1.5Si-0.6Cr, wt.%) is 116 J, but the energy for the same alloy without Q&P treatment is only 41 J; 2) the impact strength of the directly quenched steel decreased below 27 J at -12 °C, while for the Q&P treated steel the temperature corresponding to 27 J is as low as -100 °C, namely much lower DBTT. Therefore, the Q&P processing can reduce the DBTT value [229]. Similar enhancement in impact strength due to Q&P processing was also reported in [230], where the impact energy at 20 °C has almost doubled after Q&P processing of the traditionally heat treated

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steel. X. Huang *et al.* [231] demonstrated that the impact toughness of the bainitic steel increased from 84 J/cm² after quenching-tempering treatment to 104 J/cm² after Q&P treatment. The enhancement in impact toughness of Q&P processed steels was ascribed to: 1) more available RA which absorbed impact energy during testing and 2) long partitioning which released local micro-stress and improved impact toughness [231]. Additionally, improved impact resistance was achieved by A.J.S.T. Silva *et al.* in cast iron through Q&P treatment [232]. Their experiments showed that higher impact energy was consumed by the Q&P treated iron than by the conventionally processed one when both of them have UTS of 1200 MPa.

Table 1.6 Values of Charpy impact energy measured on various Q&P steel. All samples are tested with section area size 10×10 mm. (T: test temperature; CVN: Charpy V-notched impact energy)

Chemical composition (wt.%)	T (°C)	CVN (J)	Ref.
Fe-0.10C-1.51Mn-1.48Si-0.3Mo	-20	40	[230]
	20	108	[230]
Fe-0.2C-2.0Mn-1.5Si-0.6Cr	-90	34	[229]
	-60	47	[229]
	-20	77	[229]
	23	116	[229]
	50	96	[229]
Fe-0.2C-2.2Mn-1.3Si-1.1Cr-0.25Mo-0.25Ni	25	130	[231]

Note: Information on the microstructure (phase volume fraction, grain size, morphology, etc.) is not provided in the table.

From this overview, it is seen that the second generation AHSSs have the higher impact toughness than the first and third generations, while the latter two have comparable resistance to impact loading. Nevertheless, it should be noted that the data from Charpy impact testing from different laboratories have low comparability due to the discrepancy from different testing condition and systems. Furthermore, the Charpy impact testing method does not truly reproduce the process which encountered by vehicles during crash accidents.

Chapter 2

Motivation and objectives

Stimulated by the requirements of decreasing car body weight, improving passenger's safety and upgrading fuel efficiency, automobile manufacturers keep paying attention to newly developed materials, especially AHSS. In the past decades, three generations of AHSS have been explored in laboratories, and some of them have already been commercialized for fabrication of vehicle components. For a steel grade targeted at automotive applications, its high strain rate behavior should be comprehensively understood. However, the vast majority of publications on AHSSs are focusing on their mechanical properties under quasi-static deformation. In the current literature, there is a significant lack of understanding about relationship between initial microstructure of AHSSs, operating deformation mechanisms, microstructure evolution in AHSSs during dynamic deformation, and impact performance of AHSSs. No standards for high strain rate tensile testing and impact testing currently exist. Data available in literature on impact performance of AHSSs widely scatter due to different testing set ups and sample geometries used in different research laboratories. Therefore, this thesis aimed to investigate in a systematic manner the microstructure evolution, high strain rate mechanical behavior and impact performance of three AHSSs belonging to three generations having different initial microstructures.

The main objectives of this thesis can be divided into three:

1. To comparatively investigate the energy absorption capability of three AHSSs under impact loading using drop weight impact testing system. For the first time, three AHSSs from three different generations are subjected to impact testing under the same conditions, that allows to comparatively study their impact performance and relate it to their microstructure and mechanisms operating during plastic deformation.
2. To quantitatively investigate the adiabatic heating phenomenon of three AHSSs subjected to impact deformation. The *in situ* measurements of the adiabatic heating allow to precisely determine the peak temperature achieved in the material upon impact and to analyze the results with respect to the microstructure and mechanisms operating during high strain rate plastic deformation of AHSSs.
3. To investigate the high strain rate tensile mechanical behavior of a Q&P steel, belonging to the third generation AHSSs. The mechanical properties and microstructure evolution of the Q&P steel under dynamic tension are thoroughly studied and special attention is laid on stability of retained austenite and its role in high strain rate tensile behavior of the grade.

Chapter 3

Materials and experimental methods

3.1 Materials

The materials studied in this work are commercial zinc galvanized DP 1180 steel (first generation AHSS), commercial 304 SS (second generation AHSS) and a novel Q&P steel (third generation AHSS). Their chemical compositions are listed in Table 3.1.

Table 3.1 The chemical composition (in wt.%) of the studied steels.

	C	Si	Mn	Cr	Ni	Al	Nb	Ti	Cu
DP	0.12	0.28	2.48	0.59	0.01	0.035	0.023	0.026	0.01
304 SS	0.08	1.0	2.0	18.0	9	-	-	-	-
Q&P	0.25	1.5	3.0	0.015	-	0.023	-	-	-

In DP steel, 0.12 wt.% carbon was added mainly for martensite strengthening and avoiding significant reduction in weldability [4]. Manganese is highly beneficial for hardenability and austenite stabilization. To prevent formation of banded microstructure, its content was limited to 2.48 wt.%. Chromium also stabilizes austenite and increases hardenability, but its content was confined to 0.59 wt.% because of cost control. Microalloying by 0.023 wt.% of niobium was done for grain refinement effect. The material

3.1 Materials

was supplied in form of 1 mm thick sheets.

Large amount of chromium was added into 304 SS to obtain excellent corrosion resistance. Nickel and manganese promote austenite stability. Nickel also helps to reduce the corrosion rate of the steel in erosive environment. Carbon was confined to low content to avoid potential intergranular corrosion problem. The extensive addition of chromium and nickel dramatically increased the cost of 304 SS compared with other AHSSs.

For Q&P steel, 0.25% carbon was added to gain sufficient retained austenite fraction and stability [70]. The 3.0% manganese provides further stability and hardenability to austenite [233]. To suppress carbide precipitation during Q&P heat treatment, the steel was alloyed by 1.5% silicon [234]. Finally, a small amount of aluminum and chromium were added for carbide suppression and hardenability enhancement according to [235,236]

The Q&P was cast in a laboratory vacuum induction furnace. The steel slabs were hot rolled to a final thickness of 2.5 mm, followed by water jets cooling to 600 °C. Then, they were transferred to a furnace for coiling simulation at 560 °C. At the last step, the steel sheets were pickled and cold rolled to a final thickness of 1 mm. The rolled steel was cut perpendicular to the rolling direction for heat treatment. The Q&P process was carried out in a reactive annealing process simulator following the thermal cycle presented in Fig. 3.1. First, the steel was heated to 850 °C and soaked for 60 s for full austenitization and then quenched to 244 °C (quenching temperature) with a cooling rate of 20 °C/s. Subsequently, the steel was reheated to 400 °C (partitioning temperature) with the heating rate of 20 °C/s and kept for 500 s. Finally, after the partitioning step, the steel sheets were quenched to room temperature at 20 °C/s [237]. The material was supplied by University of Ghent.

Fig. 3.1 Schematic illustration of the Q&P heat treatment cycle applied to the studied steel. The $M_s = 320$ °C and $M_f = 200$ °C were determined earlier by dilatometry measurements [238].

3.2 Mechanical testing

3.2.1 Static tensile testing

Dog-bone tensile samples with a gauge length of 25 mm and a gauge width of 6 mm (according to the ASTM standard [239]) were machined via electric spark cutting along the rolling and transverse direction. Their geometry is shown in Fig. 3.2. Tensile tests were carried out using a universal electromechanical testing machine (Instron 3384) with a constant crosshead speed of 1.5 mm/min, which corresponds to the initial strain rate of $\sim 10^{-3} \text{ s}^{-1}$. Strain hardening exponent n was calculated from tensile testing data using Hollomon equation: $\sigma = K\epsilon^n$, where σ is true stress, ϵ true strain and K constant.

Fig. 3.2 Geometry of tensile samples.

3.2.2 High strain rate tensile testing

Split Hopkinson bar system has been widely used to perform high strain rate testing on subsize specimens. Usually, Split Hopkinson bar systems have two long slender bars. By striking the end of one bar, the generated stress wave transmits through this bar and reaches the specimen. Once the specimen is loaded, the wave separates into two parts, one passes through the specimen and propagates through another bar, while the rest wave reflects back to the stricken bar. The stress and strain are obtained after analyzing the waves [240]. The technique was initially designed for compression, and was subsequently upgraded for high strain rate tension and torsion experiments.

To explore the tensile mechanical behavior of Q&P steel at high strain rates, a split Hopkinson tensile bar (SHTB) was used in this work. It is shown schematically in Fig. 3.3a. The setup basically consists of two aluminum bars with a diameter of 25 mm, i.e. the input bar and output bar with lengths of 6 m and 3.125 m, respectively, between which the sample is fixed. Before a test, the impactor is accelerated towards the anvil at the free end of the input bar, thus generating an incident tensile wave in the input bar. The incident wave propagates along the input bar towards the sample and gives rise to a high strain rate deformation of the sample. By adjusting the impactor speed, the strain rate in the sample can be varied in the range of 100~5000 s⁻¹. In the present work, strain rates from ~500 s⁻¹ to ~1000 s⁻¹ were imposed. At least two specimens were tested for each condition, and the results were found to be reproducible. The sample geometry is shown in Fig. 3.3b. It should be noted that the SHTB test technique, including the specimen geometry, is not standardized. Therefore, the geometry and dimensions were chosen based on considerations formulated by P. Verleysen *et al.* in [241].

Fig. 3.3 (a) Schematic presentation of the SHTB system [241]. (b) The geometry of specimens for dynamic tensile test.

The evolution of local strain during static and dynamic tensile tests was characterized using digital image correlation (DIC) technique. Prior to testing, black speckles were applied to the white painted specimen surface to generate a random black-white pattern. During testing, the deforming speckle patterns were recorded by two high speed cameras (FASTCAM Mini AX200, Photron) operating at 30,000 frames per second. The spatial resolution of the recorded images was 256×624 pixels. From the images the strain fields on the sample surface were calculated using the commercial DIC software Vic-2D (Correlated Solutions Inc.). The subset and stepsize were 9 pixels and 1 pixel, respectively. An algorithm named normalized sum of squared differences (NSSD) was selected to obtain the strain value.

3.2.3 Drop weight impact testing

To investigate the impact resistance of AHSSs, a drop weight impact testing system (INSTRON CEAST 9350) was employed. For quasi-static punch testing in the same mode, a universal electromechanical testing machine (INSTRON 3384) was used. The $50 \times 50 \times 1$ mm samples were cut for both drop weight impact and quasi-static punch testing (Fig. 3.4b). The same hemispherical punch with a diameter of 16 mm was employed for both types of testing. The punch is made of steel having a hardness of 60

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HRC. During quasi-static punch testing, the sample was fixed effectively by a house-made holder with bolts, while in drop weight impact testing the specimen was clamped by a pneumatic system (under pressure of 6 bars), as shown in Fig. 3.4a. The shape and size of the holder and fastening components used in both kinds of testing were the same. A multipurpose lubricant (Super Lube, Syncolon®) was applied to the punch surface to minimize frictional dissipated energy. The purpose of limiting these foregoing conditions is to obtain biaxial stress mode (nearly) in the center area of the specimen in both cases. The drop weight varied in the range of 4.3~9.8 kg depending on the impact energy. The applied impact energy and drop speed were in the range of 30~140 J and 3~7 m/s, respectively, depending on the drop weight and height before test. To measure the adiabatic heating during drop weight impact, additional tests with a K-type thermocouple welded on the center of square shaped specimens were carried out, and the temperature – time plots were recorded during testing with frequency of 50 kHz.

Fig. 3.4 (a) Schematic diagram of drop weight impact and quasi-static punch tests, sectioned by a quarter. (b) Geometry of specimen and punch used in both drop weight impact testing and quasi-static punch testing.

3.3 Microstructure characterization and fracture surface analysis

3.3.1 Scanning electron microscopy (SEM)

Electron microscopy technique obtains information via detecting signals from the interaction between the electrons and specimen. The wide range of signals generated

from the specimen irradiated with incident electron beam are summarized in Fig. 3.5. These signals are frequently collected and deciphered in electron microscopy, providing enormous details about the inspected specimens. Scanning electron microscopy (SEM) belongs to the big family of electron microscopy and has been used widely in materials science research for more than half a century. In SEM, secondary electrons were mainly analyzed to obtain morphology information because secondary electrons are sensitive to the sample surface. In this work, a field emission gun (FEG) dual beam microscope (Helios NanoLab 600i, FEI) operating at a voltage of 15 kV and a current of 0.69 nA was employed to characterize the sample surfaces.

Fig. 3.5 Signals generated when a high-energy electron beam interacts with a thin specimen [242].

3.3.2 Electron backscatter diffraction (EBSD)

Electron backscatter diffraction (EBSD) is a SEM-based technique and provides the quantitative information about crystallographic orientation and phase identification for the examined specimen. The specific phase and the orientation within the small volume irradiated by electron beam were identified via indexing the Kikuchi patterns formed by the diffraction of backscattered electrons. Valuable information, such as texture, grain

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boundary, local misorientation etc., can be extracted from the orientation data. During measurements, samples are usually tilted to 70 degree so that backscatter electrons can escape from the specimen surface and generate diffraction patterns on detector, as schematically shown in Fig. 3.6.

Fig. 3.6 Schematic presentation of the EBSD technique [243].

In this work, EBSD characterization was carried out on a FEI Helios NanoLab 600i field emission gun scanning electron microscope (FEG-SEM). Kikuchi patterns were recorded by a NordlysNano detector controlled by the Aztec Oxford Instruments Nanoanalysis software (version 4.2®). A voltage of 18 kV, a current intensity of 2.7 nA and the step size of 35~800 nm were used in EBSD analysis. Orientation data were post-processed and analyzed by HKL Channel 5 software (version 5.1®). Kernel average misorientation (KAM) maps were calculated with respect to the third nearest neighbors using the HKL software. The KAM maps allow to analyze distribution of plastic microstrain over the microstructure by means of calculating the orientation difference between point clusters [244]. Histograms of statistical distribution of plastic microstrain in the analyzed microstructure were generated based on the KAM map for the analyzed specimens.

3.3.3 Transmission electron microscopy (TEM)

Transmission electron microscopy (TEM) is a type of microscopy which collects the signal from the 'transmitted' electrons. High energy electron beam accelerated by voltage 100~500 kV is commonly used to pass through thin TEM specimens. To gain a resolution

to atomic level, several magnetic lenses were employed to focus the incident beam to less than 1 nm in diameter or even smaller. In TEM imaging, the bright (BF) and dark field (DF) images are formed using direct electron beam and diffracted electrons. For selected area electron diffraction (SAED), the pattern is from diffracted electrons but using different imaging modes.

In this thesis, a FEG S/TEM microscope (Talos F200X, FEI) operating at 200 kV was employed to conduct TEM examination of the as-received and tested samples. Observations were made in both bright and dark field imaging modes. Convergent beam (CBED) and selected area electron diffraction (SAED) patterns were recorded from the areas of interest. Additionally, X-ray energy-dispersive spectrum (EDS) mapping analysis was carried out using a nanoprobe in scanning TEM (STEM) mode.

3.3.4 X-ray diffraction (XRD)

X-ray diffraction (XRD) is a technique using the diffraction phenomenon of X-ray in materials to obtain knowledge about crystal structure and lattice parameter. Important information, such as phase identity, phase fraction, texture and residual stress are frequently derived from XRD measurement. The XRD is based on the famous Bragg law,

$$n\lambda = 2d \sin \theta \quad (3.1)$$

where n is positive integer, λ X-ray wavelength, d the spacing of crystal planes and θ the angle between the X-ray and crystal planes. The diffraction of a crystal is illustrated in Fig. 3.7. Diffraction happens only when crystallographic planes meet the conditions required by Bragg law. As the planes satisfying the criteria are limited and unique for each polycrystalline material, the tested material can be identified by comparing the measured intensity-angle spectrum with the standard ones. Furthermore, the diffraction intensity of X-ray is influenced by the fraction of each phase in a matter having multiple phase, which is the foundation for phase content analysis. In this work, quantitative analysis of phase fractions was carried out on a diffractometer (Empyrean, PANalytical)

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with Cu k_α source operating at 45 kV and 40 mA.

Fig. 3.7 Schematic showing diffraction of X-rays by a crystal [245]. A, B, C,... are atoms on crystal planes. XX' and YY' are wave fronts of incident X-ray beam and diffracted beam.

3.3.5 Sample preparation for microstructure characterization

After drop weight impact and punch testing, the dome-shaped tested samples were cross-sectioned through the center, as shown schematically in Fig. 3.8. The true plastic strain accumulated at the top of the dome was determined as [246]:

$$\varepsilon = \ln \frac{h_o}{h_f} \quad (3.2)$$

Where ε is the true plastic strain, h_o the initial thickness and h_f the local thickness after testing, respectively, as shown in Fig. 3.8.

Fig. 3.8 Schematic presentation of a cross-sectioned sample after drop weight impact/punch testing. The area marked by red square refers to the region selected for EBSD and TEM characterization.

To reveal the microstructure of the materials, samples for EBSD characterization were prepared following the standard metallography grinding and polishing procedures using

colloidal silica suspension (OP-S, Struers®) at the final stage. The region selected for EBSD and TEM characterization is marked by red quadrat in Fig. 3.8.

To estimate the volume fractions of phases in DP steel, samples were polished with 0.25 µm paste at the last step and etched using 12% Na₂S₂O₅ water solution. A systematic manual point counting method according to ASTM [247] was used to determine the phase fraction on SEM images of etched sample.

For Q&P steel after dynamic SHTB testing, the positions of EBSD scanned areas are schematically shown in Fig. 3.9. Specimens were thinned to the half of thickness to avoid the influence of local strain non-uniformity. The polishing procedure was the same as mentioned before. The true strain ε in the EBSD measured position was calculated using the following equation:

$$\varepsilon = \ln \frac{A_o}{A_f} \quad (3.3)$$

where A_o is the original cross section of the gauge part and A_f the local cross section after plastic deformation.

Fig. 3.9 Schematic presentation of the areas (marked by red squares) scanned by EBSD and the measurement of width and thickness after SHTB testing. The spacing between different areas was about 900 µm, and the first area was about 1 mm away from the fracture surface.

For the as-received materials, thin TEM foils were prepared on a TenuPol 5 (Struers®) using twin-jet electron polishing method and commercial electrolyte A2. For the tested samples, TEM lamellae were milled out using focused ion beam (FIB) technique in a FIB-

3.3 Microstructure characterization and fracture surface analysis

FEGSEM dual-beam microscope (Helios NanoLab 600i, FEI) through the following steps: 1) Platinum (Pt) was deposited for the protection purpose using electron beam at 5kV and 0.69 nA then using ion beam at 30 kV and 0.23 nA; 2) One hole/trench with size of 20×30×15 μm on each side of the lamella were milled using ion beam with current of 0.23~47 nA at 30 kV; 3) The lamella was tailored and pre-thinned to the size of 10×10×1.2 μm ; 4) The lamella was transferred to a TEM copper grid using the needle in the microscope; 5) The lamella was thinned to the thickness ≤ 100 nm. Depending on the sample shapes after testing, different positions were chosen for TEM characterization. For specimens after drop weight impact testing, the positions of TEM sample are the same of EBSD characterization as shown in Fig. 3.8. For SHTB tested samples, TEM lamellae were taken from the area close to fracture.

3.3.6 Fracture surface reconstruction

A detailed quantitative analysis of fracture surfaces formed after cracking of samples tested with impact energy of >90 J was performed for DP steel by employing a SEM-based microscopic surface reconstruction technique [248–250]. This microscopic topography technique can be briefly described as following: a pair of SEM images, which were eucentricly tilted to different angles (0~10°), were used to create a 3D digital elevation model (DEM) by an algorithm which quantifies the surface height variation via searching and comparing the homologous points in the image pair [250]. Thus, one more dimension (depth, z axis) is granted to the two dimensional image, providing the possibility to measure dimple depth. In present study, SEM image pairs consisting of 1536×1103 pixels were taken at 0° and 5° tilting. A commercial MeX software (Alicona) was used to reconstruct the DEMs of fracture surfaces and for their further quantitative analysis. The fracture surface profiles were extracted from the obtained DEMs and the depth of at least 40 dimples was measured. The outcomes of these measurements were used for estimation of the energy consumed for formation of fracture surface during impact.

Chapter 4

Results and discussion

In this chapter, the obtained experimental results are presented and discussed. It consists of four subsections, which were/will be published in the following four articles:

Subchapter 4.1 corresponds to “P.K. Xia, M. Sanchez Palomar, I. Sabirov. Adiabatic heating and energy absorption capability of an advanced high strength steel during drop weight impact testing. *Materials Science and Engineering: A*. 785 (2020) 139382.”

Subchapter 4.2 corresponds to “P.K. Xia, F. J. Canillas Rodríguez, I. Sabirov. Microstructure evolution and adiabatic heating during dynamic biaxial deformation of a 304 stainless steel. (Accepted by *Materials Science and Engineering: A*)”

Subchapter 4.3 corresponds to “P.K. Xia, I. Sabirov, J. M. Molina-Aldareguía, P. Verleysen, R. Petrov. Mechanical behavior and microstructure evolution of a quenched and partitioned steel during drop weight impact and punch testing. *Materials Science and Engineering: A*. 737 (2018) 18-26.”

Subchapter 4.4 corresponds to “P.K. Xia, F. Vercruysse, R. Petrov, I. Sabirov, M. Castillo-Rodríguez, P. Verleysen. High strain rate tensile behavior of a quenching and

Results and discussion

partitioning (Q&P) Fe-0.25 C-1.5 Si-3.0 Mn steel. *Materials Science and Engineering: A*. 745 (2019) 53-62.”

4.1 Adiabatic heating and energy absorption capability of DP steel during drop weight impact testing

4.1.1 Microstructure and tensile mechanical properties of the as-received DP steel

The microstructure of the as-received material is shown in Fig. 4.1. Martensite and ferrite are the main microstructural constituents. Martensite has lathy and blocky shape (marked by blue and yellow arrows in Fig. 4.1b), while ferrite has quasi-equiaxial grains (selectively marked by red arrows in Fig. 4.1a). The volume fraction of ferrite is $25.5 \pm 4.5\%$. The spatial distribution of both martensite and ferrite is random, and the grain size of ferrite is in the range of $\sim 1 \mu\text{m}$ – $\sim 5 \mu\text{m}$ (Fig. 4.1). There is also a very low amount of ultra-fine grained austenite (0.23% as determined by EBSD), as marked in green color in Fig. 4.1b. It should also be noted that the as-received sheets are coated by a very thin hot dip galvanized zinc layer (Fig. 4.1c), having a thickness of $15.5 \pm 1.6 \mu\text{m}$. Both retained austenite and zinc layer have a negligible effect on the mechanical properties of the material, so they are not considered further in this work.

Fig. 4.1 Microstructure of the as-received DP 1180 steel: (a) typical SEM image of microstructure (ferrite is marked by red arrows) of the as-received material; (b) band contrast map (austenite is marked by green color); (c) SEM image of the coated zinc layer on DP steel obtained using backscatter electron (BSE) detector.

4.1 Adiabatic heating and energy absorption capability of DP steel during drop weight impact testing

The basic mechanical properties of the studied DP 1180 steel are summarized in Table 4.1. The steel has YS of 1062 MPa, UTS of 1244 MPa, elongation to failure of 8.2% and strain hardening exponent of 0.1, featured with ultra-high strength and limited ductility.

Table 4.1 Mechanical properties of the studied DP steel (YS: yield strength, 0.2% proof stress, UTS: ultimate tensile strength, EL_u : uniform elongation, EL_t : total elongation, n : work hardening exponent, K : constant).

YS / MPa	UTS/ MPa	EL_u / %	EL_t / %	n	K
1062±16	1244±19	4.2±0.3	8.2±0.2	0.10±0.002	1775±26

4.1.2 Drop weight impact response of the DP 1180 steel

The force-displacement curves recorded during drop weight impact testing with different impact energies are presented in Fig. 4.2a. It is seen that the maximum force increases with increasing impact energy, and all curves from tests with impact energy of ≤ 90 J have similar shape. The force linearly decreases to zero after reaching the peak value. No cracks were observed in samples tested with impact energy of ≤ 90 J. A clear swing phenomenon is observed on the curve for the specimen impacted with 100 J energy, which can be related to the formation and growth of crack. Additional tests were carried out with the impact energy of 95 J, which also resulted in formation of (micro)cracks. Therefore, it can be concluded that the drop weight impact resistance of the 1 mm thick DP 1180 steel is 90 J.

The true plastic strain values at the top of dome are plotted vs. impact energy in Fig. 4.2b. A clear linear relationship is seen with R-square of 0.99 obtained after fitting. The maximum true strain reaches up to 81.1% (at 90 J impact energy), revealing high formability of the material in high strain rate deformation under equi-biaxial stress mode.

Fig. 4.2 (a) Typical force-displacement curves recorded during drop weight impact testing of the DP 1180 steel with varying impact energy. (b) The relationship between true strain at the top of dome and impact energy. (c) The temperature variation with time recorded by thermocouple during impact testing (90 J). (d) The peak temperature measured during drop weight impact testing vs. impact energy. (e) Force-time curves for drop weight impact testing.

An adiabatic thermodynamic environment can be presumed during drop weight impact tests because of the very short deformation time (< 2 ms). Fig. 4.2c illustrates the outcomes of *in situ* temperature measurements carried out during drop weight impact testing with the impact energy of 90 J. From the curve, it is seen that the temperature at the top of the dome surges to the peak value of ~ 180 °C within ~ 50 ms followed by its steady decrease down to room temperature within ~ 5 s. However, it should be noted that the deformation time in the experiment does not exceed 1.5 ms, as shown on the load-impact time curve (Fig. 4.2e) from the given experiments. This time lag can be ascribed

4.1 Adiabatic heating and energy absorption capability of DP steel during drop weight impact testing

to (1) heat transfer from the DP steel to the welded thermocouples, and (2) a thermal inertia of thermocouple itself [251]. The peak temperature tends to increase with increasing impact energy (Fig. 4.2d) due to higher amount of plastic deformation induced into sample (Fig. 4.2b).

4.1.3 Microstructure evolution during drop weight impact testing

A thorough microstructural analysis of the material before and after testing was performed. Fig. 4.3 illustrates typical KAM maps of the material before and after impact testing with 90 J energy. In the as-received material (Fig. 4.3a), blue regions (indicating near-zero misorientation angle) correspond to the non-deformed ferrite, while the areas in green color (indicating misorientation of 2–3°) correspond to martensite. The fraction of the non-indexed pixels is low (9.5%). Impact testing with 90 J significantly increases the local misorientations all over the microstructure (Fig. 4.3b) and the fraction of non-indexed pixels (to 48.7%), indicating significant accumulation of lattice defects. The statistic KAM distribution is presented in Fig. 4.3c, evidently showing the shift of the curve from lower to higher KAM angles.

Fig. 4.3 KAM maps of (a) as-received material and (b) the sample impacted with 90 J energy. The white spots in (a) and (b) are non-indexed points. (c) Statistic distribution of KAM maps.

Fig. 4.4a and b shows the inverse pole figure (IPF) maps of the specimens before and after drop weight impact, respectively. It is seen that the microstructure of the as-received DP steel consists of equiaxial grains with diameter of up to $\sim 6\ \mu\text{m}$ (Fig. 4.4a). Biaxial stretching combined with plastic bending during impact testing results in flaser-shaped grains with average aspect ratio of 0.46. Orientation gradients in the interior of individual elongated grains can be noticed, as shown in the magnified IPF map in Fig. 4.4c. The misorientation profiles along the black lines in Fig. 4.4c are plotted in Fig. 4.4d, where low angle grain boundaries (having misorientation $<15^\circ$) can be clearly seen demonstrating the formation of substructures in the grain interior.

Fig. 4.4 Inverse polar figure (IPF) map of (a) the as-received material, (b) sample tested with 90 J energy and (c) magnified IPF map of the tested sample. Non-indexed areas are marked with white. (d) Misorientation profiles along the black lines drawn in (c).

The microstructure evolution during impact loading under biaxial stress was investigated in more detail by means of TEM. Typical TEM images of the as-received material are shown in Fig. 4.5. The martensite laths having a width of 100~300 nm, and ferrite, which has blocky shape, are clearly seen (Fig. 4.5a). Microalloying by Nb also

4.1 Adiabatic heating and energy absorption capability of DP steel during drop weight impact testing

results in formation of nanoscale spherical NbC precipitates having a size of 10~20 nm, though their volume fraction is very low (Fig. 4.5b). Their presence was confirmed by analysis of CBED patterns, as shown in inset in Fig. 4.5b, where a spot corresponding to the NbC precipitate is marked by red circle. Local EDS analysis also detected Nb segregations on those nanoprecipitates (Fig. 4.5c). The sample after impact testing shows markedly different microstructure. It is characterized by presence of dislocation tangles and irregular dislocation cell structure in the interior of individual grains (Fig. 4.5d). Size of cells is in the range of 0.3~1.0 μm , which is in a good accordance with the outcomes of the EBSD analysis (Fig. 4.4c and d). As reported previously, cell substructure can form at only 2% of true plastic strain in DP steel under uniaxial stress mode [252]. And the strain distribution inside the martensite-ferrite microstructure is normally inhomogeneous because of the higher hardness of martensite compared to ferrite. As in the present study, the accumulated plastic strain reaches up to 81.1% in the 90 J impacted specimen (Fig. 4.2b), well pronounced cell substructure is formed at such high plastic strain. Its formation can be related entirely to high strain rate deformation upon impact, while the effect of adiabatic heating on the microstructure can be ruled out due to low maximum homologue temperature (which reaches just 182 °C as seen in Fig. 4.2d) and very short time at the peak temperature (which does not exceed 50 ms, Fig. 4.2c and e). The increased KAM angle in the impacted specimen suggested the higher amount of accumulated local deformation, mainly derived from generation of dislocation multiplication as dislocation glide is the major deformation mechanism.

Fig. 4.5 (a) TEM image of the as-received material (general view). (b) TEM image of the NbC (marked with red arrows) in the as-received material. The inserted image is a convergent beam electron diffraction pattern, in which the red circle highlighted spot corresponds to the NbC, the blue rectangle and green parallelogram match the diffraction patterns of bcc iron from zone axis of [013] and [012]. (c) The corresponding EDS map of area (b) showing the segregations of niobium. (d) TEM image of the sample after 90 J impact test.

4.1.4 Fracture surface analysis

To understand failure of the DP steel under impact loading, fracture surface of cracks formed after testing with >90 J energy was carefully examined. Fig. 4.6a shows typical SEM images of 95 J impacted specimen. It is seen that the cracks show ductile fracture surface with well pronounced dimples (Fig. 4.6a). Dimples with size varying in the range of 1~6 μm are homogeneously distributed over the fracture surface, suggesting the failure

4.1 Adiabatic heating and energy absorption capability of DP steel during drop weight impact testing

process consisting of void nucleation, growth, and coalescence. It is not possible to distinguish the origins (martensite or ferrite) of the dimples because they are homogenous from an overall view despite the local difference. This means there is excellent deformation compatibility between hard martensite and soft ferrite, and both of them served as nucleation sites under equi-biaxial stress mode. The hardness difference between martensite and ferrite should be compensated by the abundant dislocations multiplied in the deformation process (Fig. 4.5d), as the increased dislocation density strengthened the ferrite phase. Some of the very coarse dimples were formed at manganese sulfide (MnS) inclusions, which acted as coarse void initiation sites leading to flat cleavage surfaces, as shown in Fig. 4.6b. As this is a commercial steel and MnS inclusions are quite difficult and costly to be completely removed when the size is below $20\text{ }\mu\text{m}$ [253], the appearance of slight MnS inclusions with a size of a few micrometers is not surprising. Their influence on deformation process and energy absorption capability of the studied DP steel will not be further discussed because of their negligible fraction.

Fig. 4.6 (a) and (b) Typical SEM images of fracture surface for 95 J impacted specimen. MnS inclusions are marked with red arrows in (b). (c) Corresponding 3D view of (a) after DEM reconstruction. (d) Fracture surface profile along the red line drawn in (a) and the measurement of dimple depth (h).

To estimate the energy consumed during fracture process, the dimple depth was quantitatively analyzed. Fig. 4.6c presents the reconstructed 3D DEM of the fracture surface in Fig. 4.6a. In Fig. 4.6d, a typical elevation (depth) profile of a dimple on fracture surface is plotted. Its corresponding position is marked by red line in Fig. 4.6a. From the profile, it is clear that the dimple has the depth of 1.2 μm . The fracture process begins with their formation and growth. The finest dimples having a depth below 0.1 μm are formed during coalescence of the coarse dimples.

The depth of dimples was statistically counted from the reconstructed DEMs. For each dimple considered, its deepest elevation value was measured even though the depth inside every dimple varies from one site to another. Dimples with diameter $>4 \mu\text{m}$ should absorb higher amount of energy than finer ones during cracking process. Over 40 dimples with size $>4 \mu\text{m}$ were analyzed, and their average depth was 1.33 μm . According to the Stüwe model [254,255], the specific energy necessary to form a unit micro-fracture surface, R_{surf} can be estimated using the following equation after approximation:

$$2R_{surf} = 2S\bar{\sigma}h_o \quad (4.1)$$

where $S = 0.25$ was adopted as the fracture surface is mainly consisted of dimples [254]. h_o is the average dimple depth and $\bar{\sigma}$ is the mean flow stress of material, described as [256]:

$$\bar{\sigma} = \sigma_{UTS} \frac{e^n}{(1+n)n^n} \quad (4.2)$$

where σ_{UTS} and n are ultimate tensile strength and the strain hardening exponent of the material (the values for RD samples in Table 4.1 were used for calculations).

The obtained R_{surf} values for the DP steel tested at high strain rate under biaxial stress mode is about 1.14 kJ/m². The estimated energy consumed for formation of the fracture surface in the 95 J impacted sample is about 2.33×10^{-3} J, and for 100 J impacted specimen, the value is about 2.23×10^{-2} J. This indicates that the energy spent for dimples formation is negligible compared to the energy spent for plastic deformation.

4.1.5 Deformation mechanisms during drop weight impact tests

The local stress state in the specimen is changing continuously during drop weight impact tests, resulting in multi-axial stress state (with tensile, shear and bending stresses etc.). For the sake of simplification, the top of the dome-shape sample was assumed to have equi-biaxial stress state. In addition, the energy spent for possible surface friction (between the punch and sample) was also neglected.

Analysis of experimental results shows a dramatic difference in ductility of the material tested at conventional strain rate in uniaxial tensile mode (~8% of strain, Table 4.1) and at high strain rate in equi-biaxial mode (~81% of true strain, Fig. 4.2b). As the drop weight impact test was completed in only a few milliseconds, and the heat generated by deformation had no time to dissipate into environment, the whole testing process can be considered as adiabatic. The adiabatic heating at the top of the dome-shaped sample reached 182 °C when tested with 90 J, which is sufficient to reduce the tensile strength of the steel. According to the previous study, the tensile strength of the DP600 steel at 200 °C decreases by 8~15% compared to the room temperature [257]. Similar results were reported for other steel grades in refs. [258,259]. However, the increase of testing temperature from room temperature to ~200 °C typically did not improve tensile ductility of DP steels [257]. It should be noted that in the case of drop weight impact testing, we have an interplay of three factors: high strain rate, equi-biaxial stress state and a temperature increase with plastic strain due to adiabatic heating effect. As dislocation glide is the only deformation mechanism in the DP steel derived from its martensite and ferrite microconstituents (Fig. 4.4 and Fig. 4.5), the flaser-shaped grains in the deformed material should be the result of dislocation glide and multiplication of different slip systems. The increased temperature and softened material, both are caused by adiabatic heating, are two promoters for dislocation nucleation and motion in the material during high strain rate deformation under equi-biaxial stress state [260]. Therefore, the softened DP steel showed better ductility under equi-biaxial stress state.

4.1.6 Energy absorption capability

The work done by the drop weight mass is transformed into two parts: (1) heat, which caused temperature change of the material and then dissipated into the environment; (2) strain energy, which was mostly stored in the material in the form of lattice defects. The first part was detected directly by the thermocouples, as shown in Fig. 4.2c and d, manifesting the fact that a lot of energy has converted into heat. The exact ratio of mechanical work transformed into heat (designated as β) has been reported previously in refs. [261–263], where Kolsky pressure tensile bar and infrared camera were used to satisfy an adiabatic condition and to catch the temperature rise during high strain rate testing, respectively. The ratio value measured experimentally is usually in the range of 0.8~0.9, which means most of the mechanical work during plastic deformation (i.e. 72~81 J in our case) is spent for material heating. Consequently, the strain energy stored during plastic deformation, i.e. the second part, consumes a smaller portion of the total work (i.e. 9~18 J). However, the strain energy, determined by the accumulated plastic strain which, in turn, is limited by the ductility of the material, is mutually influenced by the total energy introduced into sample during drop weight impact testing. This is clearly seen from Fig. 4.2b, where plastic strain accumulated by sample has a linear dependence on the impact energy. Of course, the work is also influenced by the strength of the material. The studied DP steel has an excellent tensile strength of >1200 MPa, though its ductility is not so attractive. However, this deficiency was enhanced to a large extent under dynamic biaxial loading, resulting in improved formability.

4.1.7 Conclusions of section 4.1

The microstructure evolution and energy absorption capability of a commercial high strength martensite-ferrite DP 1180 steel during drop weight impact testing were investigated. The following main conclusions can be drawn:

1. Intensive adiabatic heating effect was detected via *in situ* temperature measurements resulting in the peak temperature of 225 °C. The drop weight impact

4.1 Adiabatic heating and energy absorption capability of DP steel during drop weight impact testing

resistance of the DP steel under present testing conditions is 90 J. The ductility of the material was improved due to interplay of high strain rate, biaxial stress state and temperature increase due to adiabatic heating.

2. Dislocation glide and formation of dislocation cell substructure is the main deformation mechanism active in the DP steel during high strain rate equi-biaxial deformation.

3. Ductile failure mode features the fracture surface of the DP steel under high strain rate biaxial stress loading and the energy consumed during cracking presents very low fraction of the total plastic work.

4.2 Microstructure evolution and adiabatic heating during impact deformation of a 304 stainless steel

4.2.1 Microstructure and tensile properties of the studied 304 SS

The material used in this work is a commercial austenitic stainless steel (304 SS) in form of sheet having a thickness of 1 mm. Its chemical composition is presented in Table 3.1. Inverse polar figure (IPF) map of the as-received material on the RD-ND plane is shown in Fig. 4.7, indicating the equiaxed grains and random crystallographic texture. The average grain size (without considering annealing twin boundaries) is 10.8 μm .

Fig. 4.7 Typical IPF map from EBSD measurements of the as-received material.

The basic tensile mechanical properties of the as-received material are summarized in Table 4.2. The relatively low yield strength (YS, 345 MPa) results from the soft essence of its austenitic matrix. However, the UTS value is nearly double of YS (826 MPa), indicating excellent strain hardening ability of the material. In addition, the total elongation and fitted work hardening exponent are as high as ~97.9% and ~0.52, respectively, benefited from TRIP and TWIP effects of austenite as discussed below. The SFE of 304 SS is about 18 mJ/m² [55], which lies in the range where α' -martensite and twins form simultaneously. Numerous experimental studies confirmed the co-existence of α' -martensite and deformation twins in the 304 SS during tensile tests [264,265]. The ductility was promoted by the volume change induced by the formation of α' -martensite and twins, which act as obstacles for gliding dislocations and also enhanced the strength

4.2 Microstructure evolution and adiabatic heating during impact deformation of a 304 stainless steel

of the material.

Table 4.2 Mechanical properties of the studied 304 SS. (YS: yield strength, 0.2% proof stress, UTS: ultimate tensile strength, EL_t: total elongation, *n*: work hardening exponent, *K*: constant)

YS / Mpa	UTS / Mpa	EL _t / %	<i>n</i>	<i>K</i> /Mpa
345±20	826±20	97.9±0.3	0.52±0.007	1839±43

4.2.2 Dynamic biaxial tension and adiabatic heating

The force-displacement curves measured during dynamic impact testing with different impact energy were plotted in Fig. 4.8a. The force gradually increased with displacement due to the strain hardening. The experiments showed high reproducibility, which is also demonstrated by the superposition of the different curves. All specimens were checked carefully after impact experiments. No (micro)cracks were found on specimens impacted with energy ≤130 J, while samples impacted with 140 J always showed a crack. The swing phenomenon (blue dash rectangle in Fig. 4.8a) observed on the load-penetration depth curve for the 140 J impacted specimen (red line in Fig. 4.8a) indicates the cracking event. It can be concluded that the impact resistance of the 1 mm thick 304 SS is 130 J. This value is higher than that of the quenching and partitioning (Q&P) steel showing the impact resistance of 110 J in our previous publication [266]. However, in another report by J.A. Rodríguez-Martínez *et al.* [267], the 1 mm thick 304 SS sheet was cracked at impact energy of ~70 J. This discrepancy is attributed to the difference in the punch geometry used (conical punch in their study), as sharper punch tip perforates the steel sheet more easily than blunt ones and reduces the absorbed energy.

The temperature-time plots measured *in situ* at the top of the domed specimens, where the stress mode is assumed as biaxial, are shown in Fig. 4.8b with respect to time (up to 500 ms). The applied impact energy and average strain rate during dynamic deformation at the top center of samples are 130 J and $\sim 3.70 \times 10^2 \text{ s}^{-1}$, respectively. The impact adiabatically heated the sample (at measured region) up to ~160 °C in the first 50

ms. After the early swelling stage, the temperature increased gradually to the peak value of (~ 180 °C) at ~ 500 ms, followed by the steady decrease due to the dissipation of heat into non-deformed part of sample, as presented in the full range temperature chart inset in Fig. 4.8b. In the two cases (Fig. 4.8b), the tests finished in ~ 2.5 ms, far less than the time needed (~ 500 ms) to reach the peak temperature due to the inertia of temperature and delay effect of its measurement [251]. Nevertheless, it is obvious that large amount of plastic work, which is usually presented by Taylor–Quinney coefficient and assumed to be ~ 0.9 , was transformed into heat in an extremely short time, resulting in pronounced temperature rise recorded by thermocouples.

The peak temperature during adiabatic heating measured at the top of domed samples along with the local true plastic strain are plotted versus the impact energy in Fig. 4.8c. The accumulated strain at the top center of specimens when impacted with 120 J and 130 J energy are 60% and 81%, respectively, indicating the excellent deformation capability of the 304 SS under dynamic biaxial stress mode. This superior plasticity comes from the interplay of dislocation multiplication, twinning and martensite transformation at biaxial tension, as discussed below in this paper. It is interesting that the peak temperature is linear with impact energy for all tests, while the similar relationship of plastic strain ceased at the energy of 120 J, where appears the turning point followed by steeper increase. The linear relationship between impact energy and peak temperature may be a result of the relatively narrow strain rate range, from 1.4×10^2 to 3.7×10^2 s⁻¹ (as listed in Table 4.3), so limited variation of the Taylor–Quinney coefficient may exist. Although it is reported that the Taylor–Quinney coefficient is influenced by both strain and strain rate in 304 SS at uniaxial tensile tests, it saturates at strain rate of 2×10^2 s⁻¹ when plastic strain exceeds 0.25, whether under tension, compression or shear, as shown by simulation in [268]. As it is seen, the strain values are all above 0.3 in this study (see Fig. 4.8c), so the effect of strain on the ratio of converted plastic work may be excluded. Therefore, it is reasonable to obtain a linear correlation between energy and peak

4.2 Microstructure evolution and adiabatic heating during impact deformation of a 304 stainless steel

temperature. The drastic variation of strain when impact energy increased from 120 J to 130 J may be attributed to the necking from local strain concentration and/or due to adiabatic heating.

Fig. 4.8 (a) Force-displacement curves during drop weight impact testing. (b) Typical temperature–time plot due to adiabatic heating in dynamic impact tests with impact energy of 130 J. The inset images show the complete temperature variation. (c) The recorded peak temperature and the true plastic strain vs. impact energy.

The average strain rates for different specimens are listed in Table 4.3, even though the strain rate is not constant during impact experiments because of the gradual deceleration of the punch after contacting the specimen. For all tests with impact energy from 60 J to 130 J, the strain rates are from 1.4×10^2 to $3.7 \times 10^2 \text{ s}^{-1}$, which lie in the scope of high strain rate. The measured increase in temperature confirmed the remarkable adiabatic heating effect (Fig. 4.8b and c).

Table 4.3 Average strain rate during drop weight impact testing.

Impact energy (J)	60	80	100	120	130	140
Average strain rate (s^{-1})	140	171	206	272	369	355 ^a

^a data obtained from linear fitting.

After impact experiments, samples were cross sectioned for the subsequent microstructure analysis. The typical section profiles of the domed samples are shown in Fig. 4.9. It is seen that the center regions (i.e. top of the dome) have a smaller thickness than other parts of the section profiles. This difference originates from the deformation disparity in terms of time and location. The punch tip contacts first the center region, so higher plastic strain is accumulated and more energy is absorbed by this local area. Therefore, the center area is most vulnerable to cracking and has the highest temperature variation. This is also the reason why this area was chosen for further thorough microstructural characterization. With increasing impact energy, the thickness of the specimen decreased gradually, which is more clear in the center parts of the deformed samples.

Fig. 4.9 The section profile of samples after drop weight impact testing with different energy. A dozen of images were used to merge the whole profile for each specimen.

4.2.3. Electron backscatter diffraction (EBSD) analysis

To analyze the evolution of the microstructure of the 304 SS during high strain rate testing, EBSD characterization was carried out on the top center of deformed specimens (as illustrated in Fig. 3.8). Fig. 4.10 shows the band contrast maps (overlaid with phase maps) for specimens after testing. Each of them is a through-thickness map composed of several EBSD measurements. The sample thickness (i.e. the map width) decreases with the rising impact energy (i.e. with plastic strain, Fig. 4.8c). Initially equiaxial grains (Fig.

4.7) are gradually deformed into elliptical ones via glide of dislocations which are accumulated at grain boundaries acting as barriers for dislocation motion. These dislocation pile-ups at grain boundaries resulted in lower band contrast. On the band contrast EBSD maps, boundaries of grains without (or with a little) α' -martensite transformation have darker gray color than the grain interior, indicating higher amount of accumulated plastic deformation. Another obvious phenomenon is the inhomogeneous distribution of α' -martensite induced by deformation. This is attributed to the dependence of $\gamma \rightarrow \alpha'$ transformation on the angle (θ) between applied stress and the normal of the habit plane [269]. As martensitic transformation proceeds through the atoms displacement on habit planes under shear stress, the angle between the load axis and habit plane has a significant influence on the driving force of $\gamma \rightarrow \alpha'$ transformation. For instance, the calculated value for the Fe-Ni alloy is $\theta=39.5^\circ$ under 1000 psi (6.9 MPa) tension [269]. Therefore, only the austenite grains with the favorable crystallographic orientation transformed into α' -martensite under equi-biaxial stress. This is also responsible for the non-uniform grain size of α' -martensite. Very small amount of ε -martensite was also detected by EBSD in all impacted specimens, as marked with red in Fig. 4.10, indicating its role of precursor and also confirming the $\gamma \rightarrow \varepsilon \rightarrow \alpha'$ transformation sequence under high strain rate biaxial stress state. Additionally, twin boundaries (marked by blue lines in Fig. 4.10) were observed in austenite grains after impact deformation, verifying the twinning process under dynamic biaxial tension.

Fig. 4.10 Band contrast map overlaid with phase map (bcc in green and hcp in red) for the 304 SS after high strain rate biaxial deformation via drop weight impact tests with different energy: (a) 60 J, (b) 80 J, (c) 100J, (d) 120 J. White points correspond to the non-indexed pixels. Twin boundaries in austenite are marked by blue lines. For each sample, several adjacent areas were measured to combine into one through-thickness map. The step size is 800 nm.

The volume fraction of α' -martensite in the top center region, was quantified by EBSD and XRD methods. The results are plotted vs. true strain in Fig. 4.11 as well as the experimental data from [270]. ϵ -Martensite could not be detected by XRD because of its low fraction (less than 1 % according to the EBSD data). The high standard deviation of true strain (wide error bars) is due to its significant variation within measured area especially for the case of the 120 J impacted specimen, which has notable changing in

thickness at top center part. Except the 60 J impacted specimen (with true strain about ~30%), XRD detected a higher fraction of martensite in comparison to the EBSD method. This is because the indexing rate of EBSD characterization is related to the crystal defects, which is strongly affected by local strain. This effect is directly observed in Fig. 4.10c and 4.11d, where many white points indicating non-indexed pixels (especially around martensite) are seen. The martensite fraction measured by XRD increases with rising true strain, reaching up to 21% at the strain of ~45%, which is higher than the value measured via magnetic method in literature [270] at the same true plastic strain. Compared with the data from ref. [270], in which martensite fraction saturates at about ~13% after the strain exceeds ~32%, no obvious saturation phenomenon was found in the present study from XRD characterization. The possible reason is that the strain rate in [270] is higher ($\sim 10^3 \text{ s}^{-1}$) than that in the present study ($3.69 \times 10^2 \text{ s}^{-1}$ at 120 J), which suppressed martensite transformation more greatly by adiabatic heating [270].

Fig. 4.11 Martensite fraction at top center area (as shown in Fig. 3.8) measured by XRD and EBSD for specimens after drop weight impact tests. In [270], martensite was detected via magnetic method and the strain rate was about $\sim 10^3 \text{ s}^{-1}$ under biaxial stress state.

EBSD analysis with smaller step size shed more light on the microstructural features. Fig. 4.12a shows a typical EBSD band contrast map of impacted sample analyzed using step size of 200 nm. As mentioned above, higher local plastic strain is accumulated at grain boundaries, indicated by the darker band contrast. Additionally, similar to the

distribution of martensite, deformation twins (marked by blue lines in Fig. 4.12a) are formed only in some grains. The reason is comparable to that for martensite transformation, deformation twinning only occurs at specific planes (i.e. mainly $\{111\}$ in fcc), when shear stress component is parallel to the twinning direction ($\langle 112 \rangle$ in fcc). Another EBSD-based approach, kernel average misorientation (KAM), is used to visualize local strain via calculating the misorientation between neighboring points. The KAM map calculated with respect to the third nearest neighbors for Fig. 4.12a is shown in Fig. 4.12b. Higher misorientation angle represents higher local plastic strain values. It is clear that martensite and its surrounding area have relatively higher KAM angle (shaded with green and yellow) than untransformed blocky austenite grains. Two reasons may account for the observed phenomenon: 1) α' -martensite is composed of high density of dislocations, resulting high orientation difference inside itself; 2) the volume expansion introduced by martensite transformation squeezed outwards the neighboring austenite grains, leading to increased strain to the adjacent region.

Fig. 4.12 (a) Band contrast map overlaid with phase map (bcc in green and hcp in red) of a local area for 100 J impacted sample. Twin boundaries in austenite are highlighted with blue lines. (b) KAM map of (a). The step size is 200 nm.

A closer checkup of microstructure after dynamic biaxial deformation was conducted on the 140 J impacted specimen by means of EBSD using step size of 35 nm, which is close to the limit of this technique. The area around a crack/micro-void near the fracture surface was selected, where a microcrack formed near ϵ -martensite and α' -martensite (Fig. 4.13a). The local strain concentration induced by phase transformation should be responsible for the crack initiation. Similar phenomenon was also reported by A. Das *et*

al. [271] for 304 SS subjected to uniaxial tensile testing. They revealed that the initiation of micro-voids was related to the decohesion of the γ - α' interface or the separation of adjacent α' -martensite [271]. Additionally, deformation twin boundaries (highlighted with blue lines) are not as continuous and straight as those of annealing twins (as shown in Fig. 4.7), which normally pass through the whole grain. The morphology of mechanical twins may be explained as follows: the already twinned area changes the orientation and stress state of its neighboring region, making it no longer meet the necessary requirements for twinning but for dislocation movement or phase transformation. Therefore, other deformation mechanisms take place near the mechanically twinned area, making the observed twin boundaries discontinuous. Similarly, the fragmentary shape of martensite (Fig. 4.13a) is probably ascribed to the coexistence of and the alteration between the three deformation mechanisms (dislocation gliding, twinning and phase transformation) for 304 SS under biaxial tension.

Fig. 4.13 (a) Band contrast map overlaid with phase map (bcc in green and hcp in red) of a local area from 140 J impacted sample. Twin boundaries in austenite are highlighted with blue lines. (b) KAM map of (a). (c) Misorientation profile along the yellow line in (a). The step size is 35 nm.

The corresponding KAM map of Fig. 4.13a is depicted in Fig. 4.13b. It is clear that the region near the crack has relatively higher KAM value ($\sim 3^\circ$), indicating the regional plastic strain derived from phase transformation. The green color also has a ‘network’ shape in martensite and its neighboring area. This ‘network’ may be the result of dislocation cells. The misorientation along the yellow line in Fig. 4.13a is plotted in Fig. 4.13c, directly demonstrating the steep orientation variation up to 60° , i.e. twin boundaries. Moreover, the width of twin plates is about 200~500 nm.

In the EBSD maps (Fig. 4.10 and Fig. 4.12), the amount of ε -martensite was found to be negligible. To gain a deeper understanding of ε -martensite during high strain rate biaxial stress deformation, transmission Kikuchi diffraction (TKD or t-EBSD), another Kikuchi pattern based technique but with higher resolution than normal EBSD, was employed. The band contrast map from TKD characterization for the 140 J impacted specimen is shown Fig. 4.14. It is clear that ε -martensite has no regular grain shape and the grain size is ranging from tens of nanometers to 1 μm , dispersing between α' -martensite and/or austenite. As an intermediate phase, its grain size and shape are greatly dependent on local deformation process (i.e. continue to grow or transform into α' -martensite).

Fig. 4.14 Band contrast map overlaid with phase map of a local area from 140 J impacted sample. Several adjacent areas were measured to merge into one by TKD with step size of 20 nm.

4.2.4 Transmission electron microscopy (TEM) characterization

The microstructure was also characterized by TEM to reveal more details. A typical TEM image for the as-received material is shown in Fig. 4.15a, composed of a partial view of several equiaxial austenite grains. The austenite structure was confirmed by the insert selected area electron diffraction (SAED) pattern taken from the region marked by red circle. Fig. 4.15b shows the typical TEM image of α' -martensite transformed from austenite in dynamic impact testing. It has no regular or lath shape as observed from EBSD measurements (Fig. 4.15). A dark field image from $(200)_\gamma$ reflection is presented in Fig. 4.15a, showing the typical microstructure of deformed austenite after impact testing. The γ -austenite has a K-S relationship with α' -martensite, as confirmed by the SAED pattern inserted in Fig. 4.15c, with the variant of $(1\bar{1}1)_\gamma \parallel (011)_{\alpha'}$ and $[011]_\gamma \parallel [\bar{1}\bar{1}1]_{\alpha'}$. The detailed image of the area marked by red rectangle in Fig. 4.15c is presented in Fig. 4.15d, exhibiting austenite twin bundles (outlined by red line). The morphology of twin bundles is similar to the results reported in [264,272] for the same material under high strain rate deformation. The width of twins ranges from ~5 nm to ~20 nm, smaller than the value measured from EBSD (Fig. 4.13c, 200–500 nm). The reason is that the EBSD has lower resolution than TEM, making these twins unrecognizable in EBSD.

Fig. 4.15 TEM bright field images of (a) undeformed 304 stainless steel and (b) after drop weight impact testing (140 J). The SAED patterns of the regions marked by red circle are inserted in (a) and (b), corresponding to γ -austenite and α' -martensite, respectively. (c) TEM dark field image of γ -austenite with $(200)_\gamma$ reflection. The SAED pattern inserted in (c) was indexed as γ -austenite (green color) and α' -martensite (red color). (d) The enlarged image of the region marked by red rectangle in (c).

Further observation on microstructure at atomic scale was carried out by employing high resolution TEM (HRTEM) technique. The typical HRTEM image of the 140 J impacted specimen is shown in Fig. 4.16a, taken from $[011]_\gamma$ zone axis, where austenite matrix, twins and stacking faults (SFs) are clearly seen. The fast Fourier transformation (FFT) diffraction pattern of Fig. 4.16a is presented in Fig. 4.16b. Carefully indexing pattern of

Fig. 4.16b, as shown in Fig. 4.16c, strongly indicates the mixed diffraction patterns of austenite matrix, deformation twins and α' -martensite, following a relationship of $(1\bar{1}1)_\gamma \parallel (1\bar{1}1)_{\text{twin}} \parallel (011)_{\alpha'}$ with the zone axis $[011]_\gamma \parallel [0\bar{1}\bar{1}]_{\text{twin}} \parallel [\bar{1}\bar{1}1]_{\alpha'}$. Obviously, the K-S relationship exists between parent austenite and α' -martensite. Fig. 4.16d and e provide a closer look of the region marked with red squares in Fig. 4.16a, showing the symmetric atom arrangement of (200) planes between austenite matrix and deformation twins. It is reasonable to assign two roles simultaneously to the occurrence of twins:

1) as intermediate phase of α' -martensite formation. The previous study showed that intersections of shear bands, such as ϵ -martensite, mechanical twins, stacking faults bundles, etc. are the effective nucleation sites for strain induced martensite [264,273].

2) As the end of the TWIP deformation process. No further transformation will happen to one twin if it does not intersect with other twin, ϵ -martensite or stacking faults, as martensite only nucleates at the intersections of shear bands [264].

Fig. 4.16 (a) HRTEM image of 304 SS after drop weight impact test (140 J). (b) Fast Fourier transformation (FFT) of (a). (c) Indexing of (b), cyan $\diamond$, blue $\circ$, and red $\blacktriangle$ are representing austenite matrix, austenite twin and bcc α' -martensite, respectively. (d) and (e) are enlarged images marked with red in (a), indicating austenite matrix and twin, respectively.

4.2.5 Interplay of adiabatic heating and deformation mechanisms: phase transformations, twinning and dislocation glide

In the present study, the top center region of the domed samples was deformed at high strain rates under biaxial stress mode. Remarkable temperature increment (up to 184.3 °C) caused by adiabatic heating was measured *in situ* by thermocouples. It is well established that the total plastic work (W) done by external force is partitioned into two parts during deformation, i.e. stored energy (E) and heat (Q):

$$W = E + Q \quad (4.3)$$

Therefore, the heat generated is a part of work, namely

$$\Delta Q = \beta \Delta W \quad (4.4)$$

where β is Taylor-Quinney coefficient. The energy transferred from plastic work heats specimens adiabatically.

$$\Delta Q = \rho C_V \Delta T \quad (4.5)$$

The total work at strain ε can be obtained by its definition.

$$\Delta W = \int_0^\varepsilon \sigma d\varepsilon \quad (4.6)$$

Then the temperature increment during deformation is

$$\Delta T = \frac{\beta}{\rho C_V} \int_0^\varepsilon \sigma d\varepsilon \quad (4.7)$$

where ρ and C_V are material density and specific heat constant, respectively. Consequently, the temperature rise during dynamic deformation is strongly influenced by the local stress σ and strain ε , which have a great relation with the hardening behavior and deformation mechanism(s) of the specific material. On the other hand, material deformation behavior is generally affected by the temperature, which is even more obvious on 304 SS [274], as SIMT (strain induced martensite transformation) and dislocation gliding are temperature sensitive [270]. Also, the critical stress for twinning was reported to be temperature sensitive according to [275]. Therefore, the temperature rise caused by adiabatic heating at high strain rates and active deformation mechanisms are mutually interrelated. As for the possible dynamic strain aging (DSA) effect, it was ignored in the present study for two facts: 1) the adiabatically increased temperature (up to 184.3 °C) is lower than the temperature required for DSA to onset, which normally is above 200 °C [276,277]; 2) the strain rate range where DSA happens is below 1 s⁻¹, which is belong to (quasi)static condition and out of the strain rate region used in this study. Below we consider each mechanism individually.

Considerable experimental studies indicated that rising temperature has repressive effect on martensitic transformation in 304 SS during static uniaxial tensile testing [270,273,278], as higher temperature is favorable for stabilizing austenite. However, substantial fraction of α' -martensite was detected in the present study by both EBSD and XRD techniques (see Fig. 4.10 and 4.12), indicating significant SIMT. The suppression effect of adiabatic heating on SIMT may be negligible, although the measured adiabatic temperature is as high as 172 °C for the specimen impacted with 130 J (see Fig. 4.8c). According to the model proposed by Olson and Cohen [273], the fraction of α' -martensite ($f^{\alpha'}$) at plastic strain (ε) is empirically described as the following formula:

$$f^{\alpha'} = 1 - \exp\{-A[1 - \exp(-\alpha\varepsilon)]^n\} \quad (4.8)$$

$$A = \bar{v}^{\alpha'} K p / (\bar{v}^{sb})^n \quad (4.9)$$

where K and n are constant, p represents the probability that a shear band intersection generates a martensite embryo, $\bar{v}^{\alpha'}$ and $\bar{v}^{sb}$ are the average volume of α' -martensite and shear band, and α is a constant depending on stacking fault energy and strain rate. The value of parameter A is determined by the stability of austenite which, in turn, is determined by its chemical composition and temperature. Several investigations [270,273,278] showed that A decreases to 0 for 304 SS under static uniaxial tension when the temperature is higher than 100 °C, which means α' -martensite is not expected to appear at temperatures above this value. However, large amount of α' -martensite is observed indeed in this work Fig. 4.11. It can be hypothesized that the biaxial stress state promoted the $\gamma \rightarrow \alpha'$ transformation process. S.S. Hecker *et al.* [264,270] analyzed their experimental data using the transformation kinetics law proposed by Olson and Cohen [273], and found that balanced biaxial tension mode enhanced the formation of martensite in the 304 SS during deformation because of more shear band intersections. In another study, H.K. Yeddu *et al.* [279] showed that multiple variant of martensite were activated under biaxial stress mode, while under uniaxial tensile only one single martensite variant was triggered in one entire grain. Therefore, it is reasonable to

conclude that the promotion effect of biaxial stress on martensitic transformation compensated the impediment caused by adiabatic heating and increasing SFE.

Dislocation motion is the basic deformation mode in 304 SS and its dissociation into partials is the onset of α' -martensite embryos formation [280]. In the present study, cell structure was found after biaxial stress deformation (Fig. 4.13b). However, the dislocation glide during adiabatic heating process at high strain rate combined with biaxial stress can be a complex phenomenon. It is generally accepted that dislocation movement is sensitive to both temperature and deformation rate [281]. For instance, the dislocation motion may even transit from thermally activated slip to viscous drag mechanism with increasing strain rate [281,282]. Experimental study on the (111) $\langle 1\bar{1}0 \rangle$ type slip of aluminum single crystals at high strain rates ($10^3 \sim 10^4 \text{ s}^{-1}$) under pure shear stress indicated the presence of viscous drag mechanism at high strain rate deformation [282]. On the other hand, higher temperature decreases the resistance of dislocation movement, accompanied with the shrinkage in material yield strength and dislocation density, as reported in DP 800 steel [283] and in oxygen-free copper [284], respectively. The investigation on copper via TEM characterization revealed that the dislocation density decreased with rising temperature at static uniaxial tensile testing [284]. Furthermore, the increased dislocation density may cause temperature rise during deformation. The investigation on Fe-3Si steel done by A. Eisenlohr *et al.* [285] revealed that the sudden temperature jump by 1 K within 0.04 s was induced by the increase in dislocation density. Limited studies of the effect of stress state on dislocation behavior can be found. Additionally, dislocations are the main inner energy preservation sites together with point defects (vacancies), thus dislocation density imposes strong impacts on the stored energy as well as Taylor-Quinney coefficient and adiabatic heating during deformation. Higher dislocation density results lower Taylor-Quinney coefficient with lower adiabatic temperature. Therefore, more research effects are needed to uncoupling the convoluted relation between dislocation slip, strain rate, stress state and adiabatic heating.

4.2.6 The effect of strain on microstructure

The microstructure under dynamic biaxial tensile loading evolves gradually with respect to the accumulated plastic strain. α' -martensite, twins and dislocations formed until the fracture of specimens. No sign of saturation was found for martensitic transformation, as shown in Fig. 4.11. However, in the Olson-Cohen model [273], $\gamma \rightarrow \alpha'$ transformation during uniaxial testing saturates after a certain amount of plastic strain, which tends to increase with testing temperature, i.e. sigmoidal shaped transformation curve, as indicated in Equation (7). Extensive experimental works confirmed this saturation phenomenon in 304 SS under static tensile deformation [270,278], while under dynamic condition, the content of martensite exhibits no such effect from available literature [270,286]. In the present study under high strain rate biaxial tension, the martensitic phase in the 304 SS measured by XRD increases with increasing true plastic strain (as shown in Fig. 4.11), indicating no such saturation and no sigmoidal shape upon plastic strain. As explained before, the biaxial stress mode possibly promoted the transformation from austenite to martensite through the deformation process until failure. Furthermore, mechanical twins were observed from all specimens impacted with different energies, as shown in Fig. 4.10, but their statistical data is not available in this study. Because currently no effective quantification method exists for twins, as EBSD is limited by its indexing rate and step size, while TEM by miniscule observation area. Other drawbacks are their inhomogeneity in distribution and size, having strong relationship with local stress condition, which is out of regulation during deformation.

4.2.7 Conclusions of section 4.2

In this work, the 1 mm thick 304 SS was deformed under high strain rate biaxial stress mode via drop weight impact testing. Adiabatic heating effect and microstructure evolution was thoroughly investigated using a wide range of characterization techniques. Based on the obtained experimental results and their analysis, the main conclusions can be drawn:

1. Considerable temperature increase (up to 184.3 °C when impacted with 140 J) was measured *in situ* by thermocouples, indicating the appreciable adiabatic heating effect under dynamic equi-biaxial loading.

2. Despite the increasing SFE with temperature, the biaxial stress state compensated the possible impediment of martensitic transformation caused by adiabatic heating .

3. Very low fraction of intermediate ε -martensite (<0.5 %) is found in the deformed material by EBSD analysis. The fraction of α' -martensite cannot be precisely detected by EBSD analysis due to relatively low indexing rate. The XRD measurements showed significant increase of α' -martensite fraction (up to ~36 %) with increasing true plastic strain (up to 0.8), showing no sign of saturation and no sigmoidal shape upon plastic strain, as the biaxial stress mode possibly promoted phase transformation until failure.

4. Mechanical twins formed during dynamic equi-biaxial tensile deformation have the form of nanotwin bundles with a width of ~5 nm to ~20 nm. Their inhomogeneous distribution in the microstructure and nanosize handicapped precise quantitative analysis.

4.3 Mechanical behavior and microstructure evolution of a Q&P steel during drop weight impact and punch testing

4.3.1 Mechanical behavior of the material during drop weight impact and quasi-static punch testing

Table 4.4 summarizes the basic tensile mechanical properties of the studied Q&P steel. The steel has YS of 821 MPa, UTS of 1267 MPa, elongation to failure of 27.5% and strain hardening exponent of 0.19, indicating excellent strength and ductility.

Table 4.4 Mechanical properties of the studied Q&P steel [237] (YS: yield strength, 0.2% proof stress, UTS: ultimate tensile strength, EL_u : uniform elongation, EL_t : total elongation, n : work hardening exponent).

YS / MPa	UTS/ MPa	EL_u / %	EL_t / %	n
821	1267	16.0	27.5	0.190

Typical views of samples after drop weight impact and static punch testing are presented in Fig. 4.17. Careful analysis using microscopy did not reveal the presence of any (micro) cracks on surface of specimens tested with impact energies of 30~110 J (Fig. 4.17a). Macro crack along rolling direction was observed only in the specimens tested with 120 J, and just in the center of the hemisphere shell. On the other hand, in quasi-static punch tests, cracks occurred on the side of the hemisphere shell (Fig. 4.17b), under shearing (incomplete biaxial) stress state.

Fig. 4.17 Typical views of samples (a) uncracked after drop weight impact test (90 J impact energy) and (b) cracked on the side after quasi-static punch test (punch speed 8.33×10^{-7} m/s). Arrows indicate the rolling direction.

4.3 Mechanical behavior and microstructure evolution of a Q&P steel during drop weight impact and punch testing

The force-displacement curves from drop weight impact and static punch testing are shown in Fig. 4.18a. For drop weight impact tests (3~7 m/s), all the curves look similar, and the load increases with increasing displacement (i.e. central deflection) and rebounds back to zero after reaching the peak load. However, the curve corresponding to the sample impacted with the maximum energy of 120 J (with a drop speed of 6.74 m/s) shows a swing phenomenon in its descending part. This observation can be related to formation of crack(s), which was seen only in the samples impacted with 120 J. Thus, it can be concluded that the studied 1 mm thick Q&P steel sheet can withstand 110 J impact energy.

Fig. 4.18 (a) Force-displacement curves from drop weight impact and quasi-static punch testing; (b) Dependence of the true plastic strain with the impact energy in samples after drop weight testing.

The true plastic strain values measured at the top of the hemi-spherical part of drop weight tested samples are presented in Fig. 4.18b. It is seen that the true plastic strain increases linearly with the impact energy. The true plastic strain in the sample impacted with 30 J was 14.0%, while for an impact energy of 110 J, the true plastic strain was 53.4%. In the samples after punch testing with speeds of 8.33×10^{-5} and 8.33×10^{-7} m/s, the achieved values of true plastic strain were 41.4% and 53.7%, respectively.

4.3.2 Microstructure evolution

The microstructure of the as-received material is shown in Fig. 4.19a, where retained austenite, tempered martensite and untempered (fresh) martensite can be identified. Two types of retained austenite are seen in the EBSD maps: large blocky retained austenite

grains and the interlath lamellar-type one. The fraction of retained austenite in the Q&P treated steel measured by EBSD was 9.6% which is less than the value measured by X-ray diffraction on the same material, 18%, reported earlier by I. de Diego-Calderón *et al.* in [237]. The difference between the volume fractions determined by XRD and EBSD is related to the presence of very thin film-type retained austenite having a thickness of 10–20 nm, which cannot be detected by EBSD [127,287].

Fig. 4.19 Band contrast map (with gray color) and retained austenite phase map (with green color) of the tested specimens as a function of true plastic strain in different conditions: (a) original non-deformed material; (b) 28.6% (4.77 m/s, 60 J); (c) 42.2% (4.06 m/s, 90 J); (d) 53.4% (6.40 m/s, 110 J); (e) 42.2% (8.33×10^{-5} m/s, punch testing); (f) 53.7% (8.33×10^{-7} m/s, punch). The horizontal plane is parallel to the RD.

4.3 Mechanical behavior and microstructure evolution of a Q&P steel during drop weight impact and punch testing

The two types of martensite, tempered martensite and untempered martensite can be identified on EBSD maps due to their difference in band contrast. As seen in Fig. 4.19a, untempered martensite is darker than tempered martensite due to higher density of lattice defects which decreases its Kikuchi pattern quality. A significant effect of impact testing on the microstructure of the material is clearly seen from Fig. 4.19a to d. The initially equiaxed grains are biaxially stretched in the RD-TD plane. The band contrast is reduced and grain boundaries become blurry (as shown in Fig. 4.19c and d) due to increased dislocation density, which can be reflected indirectly from kernel average misorientation (KAM) (see Fig. 4.21).

The volume fraction of retained austenite in samples after drop weight impact testing tends to decrease with increasing true plastic strain, from the initial 9.6% to a saturation value of ~0.7% after reaching a true plastic strain of 42.2% (corresponding to an impact energy of 90 J). The volume fraction of retained austenite as a function of true plastic strain follows an exponential function with a correlation coefficient of R-Square= 0.99, and the fitting parameters presented in Fig. 4.20. Based on the fitting curve, it can be hypothesized that 0.58% of retained austenite would remain untransformed with further increasing strain.

Fig. 4.20 Relationship between volume fraction of retained austenite and true plastic strain in the samples after drop weight impact and punch testing.

The microstructure of the samples after punch testing (Fig. 4.19e and f) is similar to that of the sample after 110 J impact testing. The volume fraction of retained austenite in the specimens tested at 8.33×10^{-5} and 8.33×10^{-7} m/s was 0.44% and 0.35%, respectively, which is close to that in the specimen after 110 J impact testing (Fig. 4.20).

Typical KAM maps for the undeformed specimen and specimen after drop weight impact testing with 110 J energy (strain, 53.4%) are shown in Fig. 4.21a and b, respectively. Histograms of distribution of misorientation in the martensite and austenite generated from the KAM maps of different specimens are compared in Fig. 4.22. It is seen from Fig. 4.21a that in the underformed material, the interior of grains in tempered martensite matrix and retained austenite has near zero local misorientation, while higher local misorientations up to $\sim 1.5^\circ$ are observed in the areas corresponding to untempered martensite and at grain boundaries due to local deformation induced by phase transformation. After drop weight impact testing, with increasing plastic strain the curves for both martensite and austenite are gradually shifted towards higher misorientations due to increasing density of defects. Local misorientations of $0.8 \sim 2.5^\circ$ prevail in the KAM map for tempered martensite in the 110 J sample (Fig. 4.21b), which might be related to formation of a cell structure in the grain interior (Fig. 4.19d) due to the very high plastic strain induced into material, 53.4%. The latter sample presented slightly lower misorientations than those for the punch tested samples, despite all of them presented very similar values of true plastic strain (Fig. 4.22a). A similar phenomenon can be found in the KAM maps of retained austenite in Fig. 4.22b as well. It should be noted that the histograms of misorientation distribution in the martensite and retained austenite of specimens after quasi-static testing are similar to those in the specimens after drop weight testing to the same plastic strain (Fig. 4.22).

4.3 Mechanical behavior and microstructure evolution of a Q&P steel during drop weight impact and punch testing

Fig. 4.21 Typical KAM maps of the Q&P steel at different conditions: (a) undeformed; (b) after drop weight impact testing with 110 J energy (strain, 53.4%). White pixels correspond to the points with low indexing reliability.

Fig. 4.22 KAM statistical distribution of (a) martensite and (b) retained austenite for samples before and after testing with different parameters.

4.3.3 Cracking

The fracture surfaces of cracked samples after drop weight impact and quasi-static punch testing were carefully examined by SEM. In Fig. 4.23(a-d) fracture surfaces of a sample after drop weight testing is presented. As can be seen from Fig. 4.23a, they are characterized by the presence of dimples, thus indicating ductile failure of the material, and elongated microcracks having a length of several hundred micrometers. The long microcracks are parallel to the rolling direction. Block-like manganese sulfide (MnS) inclusions were detected by EDS inside the microcracks (Fig. 4.23b). It is well known that MnS inclusions serve as nucleation sites and propagation pathways for long microcracks in steels [288].

Fig. 4.23 SEM fracture surface of cracked specimens tested at different conditions: (a-d) after drop weight impact with 120 J; (e, f) after quasi-static punch test with speed of 8.33×10^{-5} m/s; (g, h) after quasi-static punch test with speed of 8.33×10^{-7} m/s.

The morphology of the dimples on the fracture surface shown in Fig. 4.23d is typical for those formed during ductile fracture. The fractography of static punch tested

specimens is shown in Fig. 4.23e-h. Samples after quasi-static punch testing at both speeds show similar fracture surfaces composed of long microcracks formed by MnS inclusions and 'dimples'. The morphology of the long microcracks is similar to that observed in the sample after drop weight impact tests. The morphology of the dimples is illustrated in Fig. 4.23f and h, where two types of dimples can be identified. First type of dimples (marked by solid circle in Fig. 4.23f) is similar to that observed on the fracture surface of specimens after drop weight impact tests (Fig. 4.23d). The second type of dimples (marked by dashed circle in Fig. 4.23f) presents smooth cleavage-like facets indicating local shearing failure. This observation can be related to the crack formation on the side of the hemisphere shell, where a shearing component is present in the stress mode (see Section 4.3.6).

4.3.4 Effect of strain rate on energy absorption capability

To compare the maximum energy absorption capability of the Q&P steel during drop weight impact and quasi-static punch testing, the area under the load-displacement curves was calculated [289], since it determines the energy absorbed in these processes. The calculation outcomes along with the corresponding true plastic strain values are listed in Table 4.5. The sample tested at 6.46 m/s was not fractured at the end of the test, so the energy given in Table 4.5 gives an under limit for the energy absorption capacity of the sample. The average strain rate was estimated as a ratio of the measured plastic strain to time. It is seen that the energy consumed by the 1 mm sheet of the Q&P steel at fracture in both static and dynamic tests is above 110 J. In the meanwhile, the values of maximum energy withstood by 304 SS and DP 1180 steels are 130 J and 90 J (see Sections 4.2 and 4.1), respectively. It is clearly seen that the studied Q&P steel shows enhanced crush resistance above that of the DP 1180 steel, though it is lower compared to the AISI 304 SS. This capability should stem from the unique microstructure of the material. The high strength tempered martensite matrix is able to accumulate a high amount of plastic deformation without any (micro)cracking (Fig. 4.22a), while the retained austenite

provides an extra ductility due to the TRIP effect (Fig. 4.19 and Fig. 4.20) as well as by bearing plastic deformation (Fig. 4.22b).

Table 4.5 Test speed, calculated energy, true strain, strain rate and sample state for both drop weight impact and punch tests.

Test speed (m/s)	Average strain rate (s^{-1})	Calculated energy (J)	True strain (%)	Sample state
8.33×10^{-7}	6.8×10^{-5}	122.83	53.7	cracked
8.33×10^{-5}	5.3×10^{-3}	130.66	42.3	cracked
6.46	384	104.67	53.4	uncracked
6.74	421	113.50	57.9*	cracked

*value obtained via extrapolation of the fitting function in Fig. 4.18b.

Another interesting observation is that the energies absorbed in quasi-static punch tests for both strain rates (123 J and 131 J) are higher than that in the drop weight impact tests (114 J), despite the strain rate being five to seven orders of magnitude higher in the latter case (Table 4.5). Thus, the ability of the material to absorb energy is lower at high strain rates (i.e. impact). This observation can be rationalized based on adiabatic heating effect. The Q&P steel is locally weakened due to the softening effect of adiabatic heating on the martensitic matrix during drop weight impact test. It is clearly seen from the force-displacement curves in Fig. 4.18a that during quasi-static punch testing a higher force (i.e. stress) is required to achieve similar displacements, thus indicating a higher strength of the material. This is also confirmed by measurements of adiabatic temperature change. The maximum temperature recorded during 110 J impact testing was 187 °C, which should reduce the tensile strength of the material by 10~15% without any increase of ductility [257,290]. In the meanwhile, the heat generated in quasi-static punch testing flows into the punch and fixing elements keeping the specimen temperature constant.

4.3.5 Evolution of retained austenite

In the present work, an exponential decrease of the volume fraction of retained austenite with increasing plastic strain during drop weight testing (i.e. in biaxial stretching) has been found (Fig. 4.20). It is well known that the stress state has a

significant effect on stability of retained austenite during plastic deformation [291–293]. Particularly, the biaxial stretching promotes austenite transformation compared to uniaxial testing [293]. Similar investigations on the evolution of retained austenite under uniaxial tensile deformation were already carried out. By employing XRD, Q. Hao *et al.* [294] measured the fraction of retained austenite in a Q-P-T steel as a function of plastic deformation under quasi-static uniaxial tension. The content of retained austenite decreased rapidly from 10.8% to less than 3.0% with increasing plastic strain to 11%, indicating that martensitic transformation occurred extensively at the early stages of deformation. D. De Knijf *et al.* [136] studied the behavior of retained austenite in a Q&P steel (with the same chemical composition and microstructure as the one in the current study) during quasi-static tensile testing. They also found an exponential decay of the volume fraction of retained austenite with increasing plastic strain. Therefore, the conclusion can be drawn that volume fraction of retained austenite decreases exponentially with plastic strain both under uniaxial and biaxial loading.

4.3.6 Effect of strain rate on strain distribution in tested specimens

The fracture surfaces of the specimens after impact and quasi-static punch testing show somewhat different morphologies. Long microcracks formed by MnS inclusions are present in both and chisel-point dimples dominating the fracture surface and indicating ductile failure. Generally, similar morphology of ductile fracture surface should indicate similar energy spent for its formation, according to the Stuwe model [254]. However, after integrating the force-displacement curves, we found that the studied Q&P steel absorbed a higher amount of energy in quasi-static punch tests, as shown in Table 4.5. The following explanation is proposed for this observation. The measurements of the radial strain distribution over the dome (as shown in Fig. 4.24), show their different character in samples after drop weight impact and quasi-static punch testing. For drop weight impact tested specimens, the strain peak appears in the top of the dome (red line in Fig. 4.24), while for quasi-static punched samples, the dome top has lower strain than its

surrounding area. This difference can be ascribed to the variation of friction coefficient with rising strain rate (i.e. contact speed). As confirmed previously by pin-on-disk wear testing [295], the friction coefficient between the puncher and specimen decreases with increasing speed. On the other hand, the friction condition can influence the strain distribution significantly during punch tests, i.e. high frictional force retards deformation in the top of dome causing less deformation there and more deformation in the unsupported areas [296,297]. A similar effect is observed in the present study. Fig. 4.24 illustrates plastic strain distribution over the dome of samples tested by both methods. It is clearly seen that the decreasing strain rate shifted the strain peak from the top (red line on Fig. 4.24) toward the edge of the hemispherical dome (green line on Fig. 4.24). Therefore, samples tested by quasi-static punch method cracked in the area which is displaced to the side of the dome, where the highest amount of plastic strain was accumulated (Fig. 4.17b).

Fig. 4.24 Typical strain distribution over the dome of specimens after drop weight impact and quasi-static punch testing. (The curves of 8.33×10^{-5} m/s and 4.06 m/s are for punch and drop weight impact (90 J) testing, respectively)

4.3.7 Conclusions of section 4.3

The mechanical behavior, microstructure evolution and failure behavior of a quenching and partitioning (Q&P) steel were investigated by employing drop weight impact testing and quasi-static punch testing. The following conclusions can be drawn:

1. In drop weight impact testing, the Q&P steel (1 mm thick) can withstand at least an

4.3 Mechanical behavior and microstructure evolution of a Q&P steel during drop weight impact and punch testing

impact energy of 110 J without cracking, with the steel accumulating a true plastic strain of 54%.

2. The Q&P steel has a slightly better energy absorption capability during quasi-static punch testing than during drop weight impact testing. This is mainly related to the softening of the material induced by adiabatic heating during impact testing.

3. The volume fraction of retained austenite decreases exponentially with increasing plastic strain during biaxial stretching, similarly to what has been reported before for uniaxial tensile deformation.

4. In drop weight impact tests, the specimens cracked at the top of hemisphere, while the failure position of specimens tested by quasi-static punch method was displaced to the side of the dome. This observation is related to the higher friction coefficient during quasi-static punch testing, which retards plastic deformation on the top of the dome and shifts the peak strain to the side.

4.4 High strain rate tensile behavior of a quenching and partitioning (Q&P) Fe-0.25C-1.5Si-3.0Mn steel

4.4.1 Mechanical properties

Two representative engineering stress-strain curves of the studied Q&P steel during static and SHTB tests at strain rates of 10^{-2} s^{-1} and 727 s^{-1} , respectively, are shown in Fig. 4.25a. In both static and dynamic conditions, no clear yield plateau is observed. Plastic hardening is limited in both conditions. After reaching the maximum flow stress, onset of necking occurs followed by specimen failure.

Fig. 4.25 (a) Typical engineering stress-strain curves from static and SHTB tests. (b), (c) and (d) are the total elongation (EL_t), yield strength (YS) and ultimate tensile strength (UTS) versus different strain rates for the studied Q&P steel.

Elongation to failure as a function of strain rate is presented in Fig. 4.25b. For all tensile tests in static or dynamic conditions, the maximum total elongation values are essentially in the same range (0.24~0.28), though the results from dynamic tests (750 s^{-1} and 1000 s^{-1}) show a higher scatter. Two explanations for the latter observation are proposed. First, it can be related to the features of testing using high speed SHTB system. Oscillation always has some influence on the results in high strain rate tensile

experiments, deriving from the extremely short interacting time (0.3–0.6 ms in this study) between the stress wave and the specimens [298]. Second, it might be related to the combined result of suppression of dislocation absorption of retained austenite (DARA) effect and the softening effect of adiabatic heating, as reported in [294]. The DARA refers to the fact that dislocations in martensite can be absorbed by retained austenite, making the martensite softer and intensifying its formability. However, the DARA effect is suppressed during dynamic loading because of lack of time for dislocation migration from martensite into austenite, which is unfavorable for the deformation ability. As a result, the total elongation will decrease when the adiabatic softening cannot compensate the absence of DARA [294].

The variations of yield strength (YS) and ultimate tensile strength (UTS) with strain rate during static and dynamic tensile tests are plotted in Fig. 4.25c and d, respectively. The YS of the Q&P treated steel shows similar values at static conditions and increases by more than 200 MPa for the case of dynamic tests ($500\text{ s}^{-1}\sim 1000\text{ s}^{-1}$). The UTS of the studied steel shows a moderate linear increase from the lowest strain rate (10^{-2} s^{-1}) to the highest strain rate (10^3 s^{-1}). The increasing strength of the Q&P steel during high strain rate deformation can be ascribed to two aspects: (1) strain rate hardening effect from dislocation gliding and (2) acceleration of TRIP effect from RA transformation. According to the widely used Johnson-Cook model [299], the deformation resistance increases linearly with the logarithm of strain rate due to insufficient dislocation glide. On the other hand, D.Q. Zou *et al.* [196] reported that austenite-martensite transformation during high strain rate deformation was accelerated by the increased number of dislocations and shear band intersections significantly promoting phase transformation. Our results clearly demonstrate that the RA fraction decreases exponentially with strain showing the TRIP acceleration effect (see Section 4.4.3). Therefore, the combined effect of dislocations and TRIP leads to enhanced strength of the studied Q&P steel at high strain rates.

4.4.2 Local plastic strain

Fig. 4.26 represents a selection of typical images taken by high speed camera during a test of a specimen with high strain rate of 511 s^{-1} from undeformed condition (at $0 \text{ }\mu\text{s}$) to final failure stage (at $433 \text{ }\mu\text{s}$). Corresponding maps of strain distribution are overlaid. It is clearly seen that before $300 \text{ }\mu\text{s}$, the axial strain field in the central gauge section of the specimen is homogeneous without any evidence of strain localization before a strain of about $\sim 17.5\%$ (Fig. 4.26c). Areas of localized strain appear on the central part of the maps with increasing time (from $300 \text{ }\mu\text{s}$) which indicates onset of necking. Further deformation is localized within the necking area (Fig. 4.26d-f). The highest value of the local strain is 68% (Fig. 4.26f), while the strain averaged over the gauge section at fracture obtained from the SHTB signals varies between 24% and 28% (Fig. 4.25b). Next to the neck, the latter values includes the strain of areas out of neck [300,301]. The corresponding engineering stress-strain curve is also presented Fig. 4.26g, where arrows indicate the time when the photos (Fig. 4.26a-f) were captured.

To analyze the strain distribution along the tensile axis during high strain rate deformation, the strain data was extracted from DIC and averaged for each cross section perpendicular to the tensile direction. The outcome of this analysis is shown in Fig. 4.27. A homogeneous strain distribution along the tensile axis over the gage length is observed until $267 \text{ }\mu\text{s}$, when reaching a strain value in the center of ~ 0.17 which is relevant to the maps of plastic strain distribution presented in Fig. 4.26. The peaks on the curve for $300 \text{ }\mu\text{s}$ (with strain about 0.22) indicate the onset of strain localization and necking, which becomes more pronounced upon further loading. Comparison of the local plastic strain evolution during dynamic and static tensile deformation did not show any significant differences in deformation behavior on the meso-scale. It is also similar to that observed during tensile deformation of TWIP steel in [300].

4.4 High strain rate tensile behavior of a quenching and partitioning (Q&P) Fe-0.25C-1.5Si-3.0Mn steel

Fig. 4.26 Axial strain distribution obtained by DIC during a SHTB test at different times: (a) 0 μs , (b) 133 μs , (c) 200 μs , (d) 300 μs , (e) 367 μs , (f) 433 μs . (g) The corresponding engineering stress-strain curve for Fig. 4.26a to f. Arrows indicate times. The sample was tested at strain rate of 511 s^{-1} .

Fig. 4.27 Typical distribution of axial strain calculated by DIC technique along the tensile axis of sample in a SHTB test. The sample was tested at a strain rate of 511 s^{-1} . The horizontal axis represents the position along the tensile direction. The strain is an average value for all data of the considered cross section.

4.4.3 Microstructure evolution

Band contrast maps in grey scale superimposed with fcc (austenite) phase map in green color for the studied steel before and after SHTB testing are shown in Fig. 4.28. In the undeformed sample (Fig. 4.28a), tempered martensite I and untempered martensite (UM, marked by red arrows in Fig. 4.28a) can be distinguished due to their difference in grey scale, where TM is brighter than UM. This results from significant difference in lattice defect density between these two kinds of martensite, and the Kikuchi pattern quality is significantly dependent on the lattice distortion. Because of partitioning process (for 500 s at 400°C), TM has a lower dislocation density than UM. Two morphologies of RA are observed on the EBSD maps of the undeformed specimen: large blocky RA grains having a size of $\sim 1\text{--}2 \mu\text{m}$ and finer lamellar-type ones. The finest RA laths formed between martensite grains have a thickness of $20\text{--}100 \text{ nm}$, and part of them cannot be observed via EBSD [127,287], as the step size of EBSD technique used in this study is 50 nm . Very thin interlath RA having a thickness of $10\text{--}20 \text{ nm}$, which cannot be detected by EBSD, is also present in the material [24]. We observed such interlath RA in TEM images as shown in Fig. 4.29. Dark field images clearly reveal the existence of very thin interlath RA grains (marked by red circle on Fig. 4.29b) between martensite laths (marked by red circle on Fig. 4.29c). Prior austenite grain boundaries can also be clearly seen in the

4.4 High strain rate tensile behavior of a quenching and partitioning (Q&P) Fe-0.25C-1.5Si-3.0Mn steel

microstructure of the undeformed sample, and most RA grains are located in these high angle boundaries ($> 15^\circ$). Earlier studies of Q&P steels using TEM have confirmed that both TM and UM are dislocation type martensite [302]. And the TEM characterization also shows that the TM has a lath thickness about $0.2\sim0.3\ \mu\text{m}$ while UM has a smaller thickness, around $0.1\sim0.2\ \mu\text{m}$ [24], as TM is formed from the first quenching and has larger parent grain size. It has also been figured out by selected area electron diffraction (SAED) that martensite laths in Q&P steel has K-S relationship $(\bar{1}\bar{1}0)_\alpha \parallel (\bar{1}0\bar{1})_\gamma$, $[001]_\alpha \parallel [011]_\gamma$ with the parent austenite as presented in the former literature [294]. It should be noted that the microstructure was found to be homogeneous through the thickness of the Q&P treated sheet.

Fig. 4.28 Typical band contrast maps (combined with RA phase maps) for the studied Q&P steel at different true plastic strain: (a) 0, (b) 11.5%, (c) 16.7% and (d) 29.9%. The specimen was tested at strain rate of $511\ \text{s}^{-1}$. Red arrows in Fig. 4.28(a) refers to untempered martensite.

Fig. 4.29 TEM images taken from the necking area close to the fracture surface of the sample tested at a strain rate of 511 s^{-1} . (a) Bright field (BF) image, (b) dark field (DF) image of interlath austenite, (c) DF image of martensite. Selected diffraction spots are marked with red color in the inserted diffraction patterns in (b) and (c), respectively.

Volume fraction of RA dramatically decreases with increasing plastic strain during high strain rate deformation (Fig. 4.28). The coarse RA blocks transform into martensite first, and only ultrafine RA grains remain in the microstructure, as shown in Fig. 4.28b–d. Quantitative analysis of RA volume fraction after high strain rate tensile testing shows that it decreases exponentially with plastic strain, which means that most RA grains transform into martensite at the early stage of plastic deformation, which is referred to as acceleration of TRIP effect (Section 4.1.1). A representative plot of RA volume fraction vs. true plastic strain is presented in Fig. 4.30. It is clearly seen that the RA volume fraction decreased from 9.5% to 2.2% with increasing true strain from 0% (undeformed) to 16%. Nonlinear fitting of the experimental results shows exponential character of their relationship with the R-Square of 0.984. A constant of 1.66 obtained from the fitting equation indicates that at least 1.66% of RA would remain untransformed regardless of the plastic strain which can be related to the very fine interlath RA having very high stability during plastic deformation [127]. Similar results were also reported by X. Yang

et al. [198] in the study of the relationship between RA volume fraction and plastic strain during dynamic deformation of a Q&P 980 steel and for the given Q&P steel subjected to drop weight impact testing [266]. TEM examination in the present study also revealed interlath austenite in the necking area close to the fracture surface after high strain rate deformation, as shown in Fig. 4.29b, demonstrating its high stability. By employing a modified SHTB system, which allows to induce the predetermined plastic strain during high strain rate testing, they could precisely measure the effect of plastic strain on the RA volume fraction via synchrotron XRD. Their results showed that after deformation to plastic strain of 15% at strain rate of 1000 s^{-1} , the RA fraction decreased from 12% to 4%, which is close to the values reported in the present study.

Fig. 4.30 Evolution of RA volume fraction with true plastic strain. Data from literature have also been plotted for comparison [136,294].

There are more publications on the effect of plastic strain on RA volume fraction in AHSSs during their static tensile deformation, several investigations have already been conducted. Hao *et al.* [294] measured the evolution of RA volume fraction in a quenched-partitioned-tempered steel after deformation to various plastic strain values using XRD technique. The RA volume fraction decreased exponentially from 10.8% to 3.0% with increasing plastic strain from zero to 11%. In another investigation on Q&P treated steel (with the same chemical composition as in the present study) by Knijf *et al.* [136], as shown in Fig. 4.30, the RA volume fraction also decreased exponentially with increasing plastic strain. Therefore, it can be outlined that the RA volume fraction in the Q&P steels

has the similar dependence on the plastic strain during both static and dynamic tensile testing, and its fraction exponentially decreases with increasing plastic strain.

The fraction of UM in the microstructure increases with increasing plastic strain. However, it is hard to distinguish between UM and TM grains in the band contrast maps of the deformed material (Fig. 4.28b-d). The UM grains have a higher hardness due to the higher carbon content. Therefore, plastic deformation is accumulated in softer tempered martensitic grains [237,303] increasing dislocation density and lattice distortion therein which, in turn, leads to low band contrast. Therefore, the difference in band contrast between fresh and tempered martensite is minimized, and it is much harder (or even impossible) to separate them using band contrast maps in the deformed material.

4.4.4 Fracture surface

In order to understand the failure behavior of the studied Q&P steel at different testing strain rates, the fracture surfaces were examined using scanning electron microscope (SEM). Fractography images of samples after static and dynamic tensile testing are shown in Fig. 4.31. It is seen that at all strain rates, the samples failed in ductile mode. Indeed, all fracture surfaces show a similar morphology consisting of small, shallow and non-uniform dimples, which were developed through nucleation, growth, and coalescence of microvoids. The dimple size was statistically measured using the linear intercept counting method (ASTM E112), and 35~45 SEM images (4000x magnification) were used to guarantee the reliability of the results. The histograms of dimple size distribution for samples tested at different strain rates are plotted in Fig. 4.32. It is clearly seen that the fracture surfaces of samples after static tensile testing (1×10^{-4} and $1 \times 10^{-2} \text{ s}^{-1}$) show somewhat coarser dimples compared to the samples after dynamic testing ($500 \sim 1000 \text{ s}^{-1}$). During low strain rate deformation, microvoids have sufficient time for growth with respect to the short time ($0.3 \sim 0.6 \text{ ms}$) during high strain rate deformation.

4.4 High strain rate tensile behavior of a quenching and partitioning (Q&P) Fe-0.25C-1.5Si-3.0Mn steel

Fig. 4.31 Typical SEM images of fracture surfaces of the studied Q&P steel tested at strain rate of: (a, b) $1 \times 10^{-4} \text{ s}^{-1}$, (c, d) $1 \times 10^{-2} \text{ s}^{-1}$, (e, f) 511 s^{-1} , (g, h) 742 s^{-1} and (i, j) 962 s^{-1} .

Fig. 4.32 Histograms of dimple size distribution on the fracture surface of the Q&P steel tested at different strain rates.

It should be noted that quasi-cleavage and brittle fracture were also reported for some Q&P processed steels [304]. The fracture surface of the Q&P steels is to great extent determined by the tempered condition of the martensitic matrix. In our case, the martensitic matrix was tempered at relatively high temperature (400°C) for relatively long time (500 s), resulting in its fracture by ductile mode. In our earlier work [68], we have thoroughly studied the fracture behavior of this Q&P treated steel. Quantitative 3D analysis of the fracture surface of the double-edge notched tensile specimens using state-of-the-art characterization techniques clearly revealed ductile fracture mode. In the meanwhile, significant fraction of fracture surface of this steel tempered at lower temperature 280°C for less than 1 s (resulting in less tempered martensitic matrix) showed quasi-cleavage character [68].

4.4.5 Conclusions of section 4.4

The high strain rate deformation behavior of a Q&P processed Fe-0.25C-1.5Si-3.0Mn (wt%) steel containing martensitic matrix and retained austenite was investigated via SHTB testing system. Tensile tests at conventional strain rates were also carried out for comparison. Analysis of mechanical properties, microstructural evolution and failure behavior was performed. The following conclusions can be drawn based on the experimental results.

1. The Q&P treated steel shows higher yield strength (by > 200 MPa) during high strain rate tensile deformation. Its ultimate tensile strength increases linearly with strain rate. The total elongation shows an opposite trend. The strength increase at high strain rates is ascribed to enhanced strain rate hardening and acceleration of TRIP effect.

2. The maps of local strain distribution generated by DIC technique indicate that the specimens are deformed homogeneously over the gage section before the plastic strain of 0.16 during both dynamic and static tensile testing.

3. EBSD characterization of microstructure shows that the RA volume fraction decreases exponentially with plastic strain during high strain rate tensile deformation, similar to the case of static tensile testing.

4. SEM examination of the fracture surface of tested samples indicate failure in ductile mode. Shallow and nonuniform dimples are the main features of specimens tested at all strain rates, and the dimple size is slightly higher in the specimens after static tensile testing due to longer time available for dimples to grow during static tensile testing.

Chapter 5

Comparative analysis of the experimental results

5.1 Impact performance of AHSSs

This Chapter focuses on comparative analysis of the experimental results obtained for all studied AHSSs. Table 5.1 summarizes their basic tensile mechanical properties and impact performance. It is seen that the DP 1180 steel has the highest YS (1062 MPa) but the lowest tensile elongation (8.2%) and strain hardening ability ($n = 0.10$). The 304 SS possesses the highest tensile ductility (97.9%) and strain hardening ability ($n = 0.52$) but insufficient YS (345 MPa) and UTS (826 MPa). The Q&P steel has the highest UTS (1267 MPa), which is slightly higher than that of DP steel (1244 MPa), and also high YS (821 MPa) and good tensile ductility (26.4%) along with the sufficient strain hardening ability ($n = 0.19$). It should be noted that the high tensile ductility of the 304 SS is obtained through the addition of high amount of alloying elements (Table 3.1), leading to much higher cost than that of the Q&P and DP steels.

It is seen from Table 5.1 that, under present impact testing conditions, the 304 SS has the best impact resistance (130J), followed by the Q&P steel (110 J) and the DP 1180 steel (90 J). When impacted with the same energy, higher plastic strain is accumulated in the DP 1180 sample than that in the 304 SS and Q&P steel. For example, when the impact energy is 90 J, the measured true strain for the DP 1180 steel is 81.1%, while the strain values for the 304 SS and the Q&P steel are comparable, i.e. 45.5% and 42.2%, respectively. This can be related to the lowest strain hardening ability of the DP 1180 steel (Table 5.1) resulting in macro-localization of plastic strain on the top of the dome. The DP 1180 steel also has the most pronounced adiabatic heating effect, followed by the Q&P steel and the 304 SS, as demonstrated by the cases of 110 J impact testing of these AHSSs (Table 5.1). When impacted with 110 J, the temperature increase (ΔT) reaches up to 202 °C in the DP 1180 steel, while for the 304 SS and Q&P steel, the ΔT -values are 121 °C and 164 °C, respectively.

Table 5.1 Summary of mechanical properties of AHSSs studied within frame of this thesis. (Gen: generation, YS: yield strength, 0.2% proof stress, UTS: ultimate tensile strength, EL_t: total elongation, n : strain hardening exponent)

Gen	Steel	YS /MPa	UTS /MPa	EL _t /%	n	Impact resistance /J	True strain impacted with 90 J /%	Temperature increase (ΔT) impacted with 110 J /°C	Deformation mechanisms
1	DP 1180	1062	1244	8.2	0.10	90	81.1	$\geq 202^a$	Dislocation glide
2	304 SS	345	826	97.9	0.52	130	45.5 ^b	121 ^c	Dislocation glide, twinning and phase transformation
3	Q&P	821	1267	27.5	0.19	110	42.2	164	Dislocation glide and phase transformation

^a The temperature increase (ΔT) of DP steel when impacted with 100 J is 202 °C. It is reasonable to assume that $\Delta T \geq 202$ °C when impacted with 110 J.

^b The value is obtained by fitting a linear equation to the true strain-impact energy data presented in Fig. 4.8c.

^c The value is obtained by fitting a linear equation to the adiabatic heating-impact energy data presented in Fig. 4.8c.

The energy absorption capability of the DP 1180 steel in impact testing was limited

due to the only deformation mechanism operating during high strain rate plastic deformation, dislocation glide (Section 4.1.3). This resulted in the higher local strain and in the enhanced adiabatic heating effect, $\Delta T = 202\text{ }^{\circ}\text{C}$ (Table 5.1). Retained austenite-martensite phase transformation taking place during high strain rate deformation in the Q&P steel (Section 4.3.2) increased strain hardening ability of the material, resulting in a lower plastic strain at the given impact energy and better ability of the material to absorb impact energy. As the SIMT effect has an exothermic nature, it should release latent heat [305]. In the 304 SS, the high strain rate deformation under impact was further 'harmonized' by much higher contribution of austenite-martensite phase transformation into plastic deformation (Section 4.2.3) combined with twinning. Both led to even higher strain hardening ability of the material, higher energy absorption capability and low plastic strain accumulated on the top of the dome at the given impact energy. The latter is relevant to that measured on the Q&P sample. No noticeable effect of twinning on the heat generation during plastic deformation was reported for a Fe-3wt.%Si steel in [285]. Extensive austenite - martensite phase transformation during high strain rate equi-biaxial deformation of the 304 SS (Fig. 4.11) should result even in a higher release of latent heat. However, the lowest adiabatic heating effect was recorded for the 304 SS ($\Delta T = 121\text{ }^{\circ}\text{C}$, Table 5.1). The general relationships between plastic deformation and generated heat were discussed above (see Section 4.2.5). The amount of heat generated due to plastic deformation is related to the work of plastic deformation by a Taylor-Quinney coefficient (Eq. 4.4), whereas the work of plastic deformation can be estimated by a simple integration of $\sigma(\varepsilon_p)$ relation (Eq. 4.6). The latter strongly depends on the yield strength of the material, its strain hardening ability and accumulated plastic strain. From Table 5.1, it is seen that the lowest flow stress - strain curve (i.e. the amount of plastic work) is for the 304 SS, followed by the Q&P steel, while the DP 1180 steel should show the highest amount of plastic work, when all materials are deformed to the same plastic strain. The adiabatic heating effect should be ranked similarly (i.e. ΔT for 304 SS < ΔT for Q&P < ΔT for DP 1180) assuming similar Taylor-Quinney coefficient for these steels, and it is well

correlated with the observed experimental results (Table 5.1). The contribution of latent heat release due to the austenite-martensite transformation is not significant. For example, in [262], the temperature increase related to the TRIP effect in a TRIP 800 steel was estimated in the range of 4.5~7 °C, when 30 % of retained austenite transformed into martensite.

It should be noted that the Taylor-Quinney coefficient is not a constant during dynamic plastic deformation of the steels [268]. Phase transformations cause variability of the Taylor-Quinney coefficient. Moreover, it is also affected by strain, strain rate and stress triaxiality. The latter parameters vary during high strain rate plastic deformation due to drop weight impact. Due to heterogeneous plastic deformation of samples and varying local stresses and local stress states, spatial variability of the Taylor-Quinney coefficient is expected. Therefore, the values of the Taylor-Quinney coefficient for the studied steels subjected to drop weight impact testing cannot be easily determined due to the mentioned above factors, and could be a topic of another study involving thermo-mechanical modelling.

A comparative analysis of all studied AHSSs (Table 5.1) shows that the Q&P steel has the best combination of tensile mechanical properties and impact resistance. Despite the AISI 304 stainless steel demonstrates somewhat better impact resistance, it cannot compete with the Q&P steel due to its very high cost related to the expensive alloying elements and their high content. Furthermore, the studied Q&P steel can accumulate higher plastic strain during quasi-static punch testing and absorb a higher amount of energy than during impact (Section 4.3.4), showing a remarkable biaxial stretching formability, which is advantageous for metal-forming operations. These results show excellent prospects in automotive industry for the Q&P steels. As it is well known, high strength combined with high formability is the main requirement for materials used for manufacturing of complex shape automotive parts such as B-pillar reinforcements and floor beams. Another advantage of Q&P steels for application in automotive sector lays

on their enhanced crashworthiness, which improves the passenger safety.

5.2 Effect of strain path on ductility of the Q&P steel

Comparative analysis of the Q&P samples after SHTB testing (Section 4.4) and after drop weight impact testing (Section 4.3) shows, that the Q&P steel is able to accumulate higher amount of plastic strain in the latter case, i.e. when tested in a equi-biaxial mode (Table 5.2). Two possible reasons are seen for this. First, somewhat higher amount of RA has transformed into martensite after drop weight impact testing with 110 J (0.70 % of RA remained) compared that after SHTB test (1.36% of RA remained) thus providing slightly higher ductility. Second, the tempered martensitic matrix has accumulated higher amount of lattice defects when deformed in equi-biaxial mode. This is clearly seen from comparison of KAM maps (Fig. 5.1) and histograms of distribution of misorientation in the martensite and austenite generated from the KAM maps of different specimens (Fig. 5.2). It is seen from that in the sample after SHTB testing, significant portion of microstructure (both martensite and retained austenite) has local misorientations below 1° , while higher local misorientations in the range of $1\sim 3^\circ$ prevail in the microstructure of the sample after drop weight impact testing, thus indicating higher density of lattice defects accumulated in both phases. It is well known that sheets metals can deform to a higher extent under biaxial tension compared to uniaxial tension as equi-biaxial deformation promotes the activation of slip on multiple slip planes.

Table 5.2 Comparative analysis of plastic strain accumulated by the Q&P specimens during drop weight impact testing and SHTB testing and remained volume fraction of retained austenite in the Q&P steel.

	True strain/%	RA fraction/%	Index rate/%
Drop weight impact test with 60 J	28.6	1.23	81.61
Drop weight impact test with 110 J	53.4	0.70	69.66
SHTB test	29.9	1.36	86.00

Fig. 5.1 Band contrast maps (combined with RA phase maps) for the studied Q&P steel after (a) drop weight impact testing (110 J, local true strain 53.4%) and (b) SHTB testing (local true strain 29.9%). (c) and (d) are KAM maps of (a) and (b), respectively. ((b) and (c) was presented previously in Fig. 4.28d and Fig. 4.21b)

Fig. 5.2 KAM statistical distribution of (a) martensite (b) retained austenite in the sample after drop weight impact testing (110 J, true stain 53.4%) and SHTB specimen (split Hopkinson bar tensile testing, true strain 29.9%). ((b) was presented previously in Fig. 4.22.)

Chapter 6

Conclusions of the thesis and future work

6.1 General conclusions

In this work, impact response, microstructure after drop weight impact testing, mechanisms operating during high strain rate deformation and the related adiabatic heating phenomenon were investigated for dual phase (DP) 1180 steel, 304 stainless steel (304 SS) and quenching and partitioning (Q&P) steel, belonging to the first, second and third generation AHSSs, respectively. Additionally, the mechanical behavior of the Q&P steel subjected to high strain rate uniaxial tensile testing was analyzed, and its microstructural response was studied. Based on the obtained experimental results and their analysis, the following general conclusions can be outlined:

1. Under present condition, the 304 SS showed the highest impact energy absorption capability (130 J) followed by the Q&P steel (110 J) and the DP 1800 steel (90 J). The highest adiabatic heating effect (225 °C, 100 J) was recorded for the DP 1180 steel followed by the Q&P steel (187 °C, 110 J) and the 304 SS (172 °C, 130 J).

2. Dislocation glide is the only deformation mechanism operating during high strain rate plastic deformation of the DP 1180 steel. The impact resistance of the Q&P steel is enhanced due to the combined effect of dislocation glide and TRIP phenomenon. These mechanisms along with deformation twinning active in the 304 SS further increased its impact resistance.

3. The Q&P steel appears to be more attractive for automotive applications compared to the 304 SS and DP 1180 steels, as it shows a good combination of excellent tensile mechanical properties, impact resistance and relatively low cost.

4. The Q&P steel shows higher ductility when tested at high strain rate via drop weight impact compared to the SHTB testing. This is related to slightly higher contribution of retained austenite-martensite phase transformation into plastic deformation and higher capability of microstructural constituents (tempered martensitic matrix and remaining retained austenite) to accumulate lattice defects when the material is tested in equi-biaxial tensile mode.

6.2 Future work

Based on the present study, the following areas of future work are envisioned:

1. The effect of stress state on ductility of the 304 SS and mechanisms operating during high strain rate uniaxial tensile deformation should be resolved. The SHTB testing should be carried out and the microstructure developed in the material should be characterized. The outcomes of the study should be compared with the existing results from the drop weight impact testing of the 304 SS. The interrupted SHTB testing to given strain combined with in situ measurements of adiabatic heating will shed more light on progress of adiabatic heating during high strain rate uniaxial testing and its interplay with microstructure evolution and phase transformation kinetics.

2. The RA in the Q&P steel was transformed into martensite rapidly with increasing plastic strain during drop weight impact testing. Improved mechanical stability of RA may enhance the performance of Q&P steel further. Additional stabilizing of RA by other elements, for instance Mn, would be of interest.

3. In this work, the microstructure and local strain was analyzed on the top of the dome-shaped samples (after drop weight impact testing) where the stress state is equibiaxial. It is suggested to carry out investigation on the side of the dome-shaped specimens where shear stress component dominates. The microstructure evolution in these regions is of great interest. In order to accurately analyze the complicated local stress/strain state, numerical simulations should be performed.

4. Thermo-mechanical modelling of the Taylor-Quinney coefficient during high strain rate plastic deformation in drop weight impact testing would be of great interest. The study could focus on the effect of austenite-martensite transformation during high strain rate plastic deformation on the Taylor-Quinney coefficient, as well as its spatial variability in the tested sheets.

5. The present work studied impact resistance of sheets using drop weight impact method. It would be interesting to investigate impact performance of thin-walled columns composed of various cross-section shapes with different size due to their similarity with automobile parts. A correlation between experimental results from drop weight impact testing of sheets and thin-walled columns would be of great interest for manufacturers of automotive parts.

Chapter 7

Publications and conferences

The following papers were published or submitted based on the obtained results obtained within frame of this PhD thesis.

1. **P.K. Xia**, I. Sabirov, J. M. Molina-Aldareguía, P. Verleysen, R. Petrov. Mechanical behavior and microstructure evolution of a quenched and partitioned steel during drop weight impact and punch testing. *Materials Science and Engineering: A*. 737 (2018) 18-26. doi: 10.1016/j.msea.2018.09.015
2. **P.K. Xia**, F. Vercruysse, R. Petrov, I. Sabirov, M. Castillo-Rodríguez, P. Verleysen. High strain rate tensile behavior of a quenching and partitioning (Q&P) Fe-0.25 C-1.5 Si-3.0 Mn steel. *Materials Science and Engineering: A*. 745 (2019) 53-62. doi: 10.1016/j.msea.2018.12.064
3. **P.K. Xia**, M. Sanchez Palomar, I. Sabirov. Adiabatic heating and energy absorption capability of an advanced high strength steel during drop weight impact testing. *Materials Science and Engineering: A*. 785 (2020) 139382. doi: 10.1016/j.msea.2020.139382

4. **P.K. Xia**, F. J. Canillas Rodríguez, I. Sabirov. Microstructure evolution and adiabatic heating during dynamic biaxial deformation of a 304 stainless steel. doi: 10.1016/j.msea.2020.139829. (Accepted by Materials Science and Engineering: A on June 23, 2020)

Other research merits.

1. **P.K. Xia**, I. Sabirov, J. M. Molina-Aldareguía, P. Verleysen, R. Petrov. Mechanical behaviour and microstructure evolution of a quenching and partitioning steel during drop weight impact tests. THERMEC'2018: International Conference on Processing & Manufacturing of Advanced Materials. July 8–13, 2018, Paris, France.
2. **P.K. Xia**, I. Sabirov, F. Vercruysse, R. Petrov, P. Verleysen. Drop weight impact resistance of advanced high strength steels (AHSSs). DYMAT Winter School 2020. February 9–14, 2020, Les Houches, France.
3. T. Sapanathan, N. Jimenez-Mena, I. Sabirov, M. A. Monclús, J. M. Molina-Aldareguía, **P.K. Xia**, L. Zhao, A. Simar. A new physical simulation tool to predict the interface of dissimilar aluminum to steel welds performed by friction melt bonding. Journal of Materials Science & Technology. 35 (2019) 2048-2057. doi: 10.1016/j.jmst.2019.05.004
4. T. Sapanathan, L.P. Ding, A. M. Bettanini, I. Sabirov, **P.K. Xia**, M. A. Monclús, J. M. Molina-Aldareguía, H. Idrissi, P. J. Jacques, A. Simar. On the formation of modified intermetallics at an Al/Fe interface via segregated Si. MSE Congress 2020. September 22–25, 2020, Berlin, Germany. (Online conference)

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