Microstructure evolution and adiabatic heating during dynamic biaxial deformation of a 304 stainless steel

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Abstract

This study focuses on the microstructure evolution and adiabatic heating during high strain rate biaxial deformation of a 304 stainless steel. Adiabatic heating is measured *in situ* during drop weight impact testing of the studied material. The considerable temperature rise induced by adiabatic heating (up to 184.3 °C when impacted with 140 J) is detected. The microstructure was analyzed by X-ray diffraction (XRD), electron backscatter diffraction (EBSD) and high resolution transmission electron microscopy (HRTEM) techniques. Dislocation glide, twinning and austenite-martensite phase transformation are operating during high strain rate plastic deformation. The XRD analysis shows a steady increase of α' -martensite fraction (up to 36 %) with plastic strain (up to 0.8), whereas the fraction of intermediate ϵ -martensite remains negligible (< 0.5 %). The decreasing stacking fault energy (SFE) with temperature along with biaxial stress compensated the possible impediment of martensitic transformation caused by adiabatic heating. Nanotwins having a width of ~5 nm to ~20 nm are inhomogeneously distributed in the microstructure. Interrelationship between adiabatic heating, deformation mechanisms and microstructure evolution is discussed.

Keywords: 304 stainless steel; dynamic deformation; biaxial stress; adiabatic heating; martensite transformation

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1. Introduction

The heat generated during high strain rate plastic deformation, which is frequently seen in both manufacturing and service processes, such as high-speed stamping, rolling, cutting, car crash accidents, etc., has little time to dissipate into the surrounding environment, resulting in temperature increase of deformed material and changing its thermodynamic state from isothermal into adiabatic. At least two possible consequences may arise due to the drastic variation in temperature during high strain rate deformation: 1) noticeable softening of material and 2) alteration of deformation mechanisms at elevated temperatures, which may promote the formation of adiabatic shear bands followed by necking. The adiabatic heating effect on microstructure and mechanical properties of metallic materials has been widely studied [1,2]. The work on a Fe–20Mn–0.6C TWIP steel revealed that the temperature increased up to ~170 °C during tensile testing at strain rate of 0.5 s⁻¹, leading to decrease of ultimate tensile strength (UTS) by ~200 MPa [2]. The increased temperature has consequently increased the stacking fault energy (SFE) by 25 mJ/m², which prohibited the twinning process during plastic deformation [2]. Therefore, elucidating the influence of adiabatic heating on performance of materials is essential for metals which may undergo high strain rate process.

The temperature rise in the adiabatic condition is mainly attributed to the plastic work done by external loading. Taylor and co-workers found the whole plastic work was not entirely converted into heating during metal deformation, and assigned a ratio (denoted as β) to assess the fraction of mechanical energy dissipated into heat [3,4], known as the Taylor–Quinney coefficient. Except the energy converted into heat during deformation, the rest portion of mechanical energy is stored inside the deformed material, which is referred to as stored energy or strain energy. As most of metal forming processes involve heat generation, a precise measurement of this coefficient is of great importance. Thorough studies of the Taylor–Quinney coefficient in polymers [5], copper [6], steel [7] etc., were carried out by employing various techniques including welded (or embedded) thermocouples [8] and high speed infrared cameras [9]. Some detailed research works showed that the Taylor–Quinney coefficient is not a constant and has a functional dependence on both strain and/or strain rate [7,9,10]. On the other hand, when evaluating or modeling the adiabatic heating effect of ductile metals at dynamic deformation processes, the β value is frequently assumed to be in the order of 0.9 or

1 for the sake of simplicity [2,11].

The coefficient β has a strong relationship with respect to the specific mechanism through which the deformation undergoes. Depending on the SFE of the metallic material, deformation at dynamic conditions proceeds singularly or cooperatively through dislocation gliding, phase transformation and/or mechanical twinning. The latter two are respectively referred to as the transformation and twinning induced plasticity effects (i.e. TRIP and TWIP), which are two excellent methods to handle the dilemma between strengthening and toughening especially in steels [12]. For the case where dislocation slip governs the plastic deformation, the Taylor–Quinney coefficient is mainly governed by the dislocation structure even though the flow stress and work hardening are controlled by the dislocation density, as simulated by Benzerga *et al.* [13] via discrete dislocation plasticity analysis. Mechanical twinning has quite different influence on the Taylor–Quinney coefficient and stores little energy of cold work. For example, experimental and numerical study on the energy dissipation in Zirconium by Padilla *et al.* [14] ruled out the significant contribution of mechanical twins on the energy storage. They found the stored energy might be partitioned in the form of deformation inhomogeneity instead of twins, even though mechanical twins contributed substantially to the strain and work hardening. The ratio of converted plastic work in the deformation process accompanied by phase transformation is more complex than that of dislocation gliding and twinning, especially for the transformation from metastable austenite to α' -martensite, during which additional heat is released known as an exothermal process. In these processes, the measured temperature rise consists of the dissipated external plastic work and the internally generated latent heat, sometimes yielding a Taylor–Quinney coefficient exceeding 1 because of the extraneous heat from phase transformation. Some detailed description involving the effect of phase transformation on the Taylor–Quinney coefficient can be found in the investigation on TRIP steel [7], 304 SS [10] and pure iron [15].

Interrelationship between adiabatic heating, microstructure and mechanical behavior of austenitic stainless steels is very complex, as dislocation gliding, twinning and strain induced martensite transformation (SIMT) operate simultaneously when responding to high strain rate loading. The 3XX series stainless steels are extensively used for energy absorption purposes, like in impact, crash and blast [16–18], involving intricate interactions between energy transfer

and microconstituents evolution. It is well established that the stability of metastable austenite has an inverse association with temperature, so the latent heat released by SIMT hinders SIMT itself, which is not the case in the deformation processes prevailed by dislocation slip or twinning. On the other hand, the contribution on inelastic deformation from dislocation gliding and twinning progressively decreases as austenite transforms into martensite. In addition, the influence imposed by strain rate on these deformation mechanisms should not be neglected when analyze the corresponding microstructure.

As a representative austenitic stainless steel, 304 SS has been extensively used in many fields like food processing, automobile parts and nuclear reactors, due to the excellent mechanical properties and distinguished corrosion resistance. During high strain rate loading, the austenite deforms via the development of dislocations, twins and martensitic phase, which are influenced by temperature variation. Therefore, the effect of adiabatic heating on the evolution of microstructure is unavoidable. For instance, the experimental study on 304 SS showed that the temperature rise caused by adiabatic heating suppressed the α' -martensite transformation at high strain rates [19]. Another investigation by means of surface mechanical attrition treatment indicated that twinning is dominant under high strain rate (10^4 – 10^5 s⁻¹), while dislocation movement and $\gamma \rightarrow \alpha'$ transformation are prevalent under lower strain rate (10 – 10^3 s⁻¹) [20]. Consequently, more research studies are necessary to thoroughly understand the evolution of microstructure during high strain rate deformation.

Except temperature and strain rate, stress state is another important factor which has significant effect on the deformed microstructure. For example, Beese *et al.* [21] revealed the remarkable influence of load triaxiality on the kinetics of SIMT by numerical simulation. Unfortunately, not many such studies can be found in the current literature. One reason for the scarcity of report is the difficulty to realize biaxial stress, much less deformation at high strain rate in biaxial stress state. As a small part of the serial work, S. S. Hecker *et al.* [19,22] studied the microstructure of 304 SS sheet under TEM after high strain rate biaxial tension testing, which was achieved through hemisphere punch with gas gun projectile. The estimated strain rate was about 10^3 s⁻¹. They concluded that more shear band intersections (stacking faults, twins, ϵ -martensite) formed under biaxial deformation than under uniaxial tension. M. Lepareux *et al.* [23] studied the deformation process of 304L SS plates subjected to high speed impact (biaxial

tension) via flat-nosed cylindrical punch at the speeds up to ~ 10 m/s. Their experiments demonstrated that a large fraction of the incident impact energy was absorbed by the plastic deformation outside the impact zone. From the above review, it is clear that the response of 304 SS under dynamic biaxial loading is not comprehensively understood yet and more relative studies are needed. The main objective of this study is to present a full evaluation of 304 SS during dynamic deformation under biaxial stress state.

2. Material and experimental procedures

2.1. Material

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 1. Inverse polar figure (IPF) map of the as-received material on the RD-ND plane is shown in Fig. 1, indicating the equiaxed grains and random crystallographic texture. The average grain size (without considering annealing twin boundaries) is $10.8 \mu\text{m}$.

Fig. 1. Typical IPF map from EBSD measurements of the as-received material.

Table 1. Chemical composition (wt. %) of the studied material.

C	Si	Cr	Mn	Ni	Fe
0.08	1.00	18.00	2.00	9.00	Bal.

2.2. Biaxial tension under high strain rates

To achieve biaxial stress mode at dynamic condition, a drop weight impact testing system (Instron CEAST 9350) was used. The hemisphere-nosed punch equipped on the system helped to realize balanced biaxial tension, as reported in [19,21]. The diameter of the punch tip and the specimen size are $\varnothing 16$ mm and $50 \times 50 \times 1$ mm, respectively. A pneumatic fastening system was used to firmly clamp the sample during impact testing. More details about the holder and fasten parts are schematically shown in Fig. 2a. To reduce the friction, the punch tip was lubricated by a multipurpose lubricant (Super Lube, Syncolon®). The total impact energy was controlled in the range of 60–150 J by changing the height of the drop hammer, which had a weight of 5.3 kg. In order to have a quantitative insight of the temperature evolution during impact tests, a K-type thermocouple was welded (spot resistance welding) on the backside of each sample in the center. The data collection frequency for temperature was 50 kHz.

The hemisphere shaped samples after impact testing were sectioned along the center for further microstructural studies as well as to measure the amount of true plastic strain accumulated by samples. The latter was calculated using equation [24]

$$\varepsilon = \ln \frac{h_o}{h_f} \quad (1)$$

where ε is the true plastic strain, h_o is the initial thickness and h_f is the thickness after testing, respectively, as shown in Fig. 2b.

a)

Fig. 2. (a) Schematic drawing of the set-up for drop weight impact testing used in this study. (b) Schematic view of a sample after drop weight impact testing. The area selected for further microstructural characterization is marked by white square in (b) [25].

2.3. Microstructure characterization

The microstructure before and after impact testing was carefully analyzed via electron backscatter diffraction (EBSD) and high resolution transmission electron microscope (HRTEM). Standard metallography procedure with final polishing by OP-S suspension (Struers®) was employed for EBSD sample preparation. The area selected for EBSD characterization of specimens after impact testing was marked by white square in Fig. 2b. A FEI NanoLab 600i field emission gun scanning electron microscope (FEG-SEM) was used to carry out EBSD characterization. TEM foil sample of the as-received material was prepared using a twin-jet electropolishing equipment (TenuPol 5, Struers®). TEM lamellar of specimens after impact testing was milled out from the EBSD analyzed area using Ga^+ focused ion beam (NanoLab 600i, FEI). TEM characterization was carried on a FEG S/TEM microscope (Talos F200X, FEI) at operating voltage of 200 KV. Transmission EBSD (t-EBSD) was carried out for deformed specimen following the procedures described in [26]. For quantitative analysis of phase fractions, X-ray diffraction (XRD) measurements were carried out on a diffractometer (Empyrean, PANalytical) with a $\text{Cu } \alpha$ source operating at 45 KV and 40 mA.

3. Results

3.1. Uniaxial tensile properties of the studied 304 SS

The basic tensile mechanical properties of the as-received material are summarized in Table 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 $\sim 97.85\%$ and ~ 0.52 , respectively, benefited from TRIP and TWIP effects of austenite as discussed below. The SFE of 304 is about 18 mJ/m^2 [27], which lies in the scope 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 [22,28]. The ductility was promoted by the volume change induced by the formation of α' -martensite and twins, which impeded the dislocation gliding as obstacles and enhanced the strength of the material.

Table 2. Mechanical properties of the studied 304 SS steel. (YS: yield strength, 0.2% proof stress, UTS: ultimate tensile strength, EL: total elongation, n : work hardening exponent, K : constant)

YS / MPa	UTS / MPa	EL / %	n	K/MPa
345 $\pm$ 20	826 $\pm$ 20	97.85 $\pm$ 0.27	0.52 $\pm$ 0.007	1839 $\pm$ 43

3.2. Dynamic biaxial tension and adiabatic heating

The force-displacement curves during dynamic impact testing with different impact energy were plotted in Fig. 3a. The force gradually increased with displacement as a result of the strain hardening. The experiments showed high reproducibility, as 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 $\leq 130 \text{ J}$, while samples impacted with 140 J showed a crack. The swing phenomenon (blue dash rectangle in Fig. 3a) observed on the load–penetration depth curve for the 140 J impacted specimen (red line in Fig. 3a) indicates the cracking event. It can be concluded that the impact crashworthiness 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 [25]. However, in another report by J.A. Rodríguez-Martínez *et al.* [16], the 1 mm thick 304 SS sheet was cracked at impact energy of $\sim 70 \text{ J}$. 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, is shown in Fig. 3b 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 energy adiabatically heated the sample (at measuring region) up to $\sim 160 \text{ }^\circ\text{C}$ in the first 50 ms. After the early swelling stage, the temperature increased gradually to the peak value at $\sim 500 \text{ ms}$, 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. 3b. In the two cases (Fig. 3b), the tests finished in $\sim 2.5 \text{ ms}$, far less than the time needed ($\sim 500 \text{ ms}$) to reach the peak temperature due to the inertia of temperature and delay effect of its measurement [29]. Nevertheless, it is obvious that large amount of plastic work, which is usually presented by Taylor–Quinney coefficient and assumed to be $\sim 90 \%$ Ref., 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. 3c. 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 $3.7 \times 10^2 \text{ s}^{-1}$ (as listed in Table 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 $2 \times 10^2 \text{ s}^{-1}$ when plastic strain exceeds 0.25, whether under tension, compression or shear, as shown by simulation in [10]. As it is seen, the strain values are all above 0.3 in this study (see

Fig. 3c), 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 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. 3. (a) Force-displacement curves during dynamic biaxial tension (drop weight impact) tests. (b) Typical temperature–time plot due to adiabatic heating in dynamic impact tests with impact energy of 130 J. The inset images show the full 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 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 3.7×10^2 s⁻¹, which lie in the scope of high strain rate. The measured increase in temperature confirmed the remarkable adiabatic heating effect (Fig. 3b and 3c).

Table 3. Average strain rate during drop weight impact tests.

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. 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. 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.

3.3. 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. 2b). Band contrast is an indicator of the quality of Kikuchi patterns which are influenced significantly by the crystal distortion and defect density. The higher the number of the band contrast, the higher the pattern quality. Fig. 5 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. 3c). Initially equiaxial grains (Fig. 1) are gradually deformed into elliptical ones via dislocation gliding 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 small part) 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 [30]. 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 [30]. Therefore, only the austenite grains with the favorable crystallographic orientation transformed into α' -martensite under quasi-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. 5, 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. 5) were observed in austenite grains after impact deformation, verifying the twinning process under dynamic biaxial tension.

Fig. 5. Band contrast map overlaid with phase map (bcc in green and hcp in red) 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 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. 6 as well as the experimental data from [19]. 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. 5c and 5d, 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 [19] at the same true plastic strain. Compared with the data from ref. [19], 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 [19] 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 [19].

Fig. 6. Martensite fraction at top center area (as shown in Fig. 2) measured by XRD and EBSD for specimens after drop weight impact tests. In literature [19], martensite was detected via magnetic method and the strain rate is 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. 7a 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. 7a) 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 location strain via calculating the misorientation between neighboring points. The KAM map calculated with respect to the third nearest

neighbors for Fig. 7a is shown in Fig. 7b. Higher misorientation angle represents bigger local plastic strain level. 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. 7. (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 were 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. 8a). The local strain concentration induced by phase transformation should be responsible for the crack initiation. Similar phenomenon was also reported by Arpan Das *et al.* [31] for 304 SS subjected to uniaxial tensile testing. They revealed that the initiation of micro-void is related to the decohesion of the γ - α' interface or the separation of adjacent α' -martensite [31]. Additionally, deformation twin boundaries (highlighted with blue lines) are not as continuous and straight as those of annealing twins (as shown in Fig. 1), which normally pass through the whole grain. The morphology of mechanical twins may be explained as this: 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. 8a) 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.

The corresponding KAM map of Fig. 8a is depicted in Fig. 8b. 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. 8a is plotted in Fig. 8c, directly demonstrating the steep orientation variation up to 60° , i.e. twin boundaries. Moreover, the width of twin plates is about 200–500 nm.

Fig. 8. (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.

In the EBSD maps (Fig. 6 and Fig. 8), 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 electron 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. 9. 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. 9. 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.

3.4. 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. 10a, 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. 10b 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. 10). A dark field image from $(200)_\gamma$ reflection is presented in Fig. 10a, 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 insetted in Fig. 10c, 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. 10c

is presented in Fig. 10d, exhibiting austenite twin bundles (outlined by red line). The morphology of twin bundles is similar to the results reported in [20,22] 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. 9c, 200–500 nm). The reason is that the EBSD has lower resolution than TEM, making these twins unrecognizable in EBSD.

Fig. 10. 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. 11a, 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. 11a is presented in Fig. 11b. Carefully indexing pattern of Fig. 11b, as shown in Fig. 11c, 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. 11d and 11e provide a closer look of the region marked with red squares in Fig. 11a, 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 [22,32]. 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 [22].

Fig. 11. (a) Atomic 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. Discussion

4.1. Interplay of adiabatic heating and deformation mechanisms : phase transformation, 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):

$$W = E + Q \quad (2)$$

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

$$\Delta Q = \beta \Delta W \quad (3)$$

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

$$\Delta Q = \rho C_V \Delta T \quad (4)$$

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

$$\Delta W = \int_0^\varepsilon \sigma d\varepsilon \quad (5)$$

Then the temperature increment during deformation is

$$\Delta T = \frac{\beta}{\rho C_V} \int_0^\varepsilon \sigma d\varepsilon \quad (6)$$

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 [33], as SIMT and dislocation gliding are temperature sensitive [19]. Also, the critical stress for twinning was reported to be temperature sensitive according to [34]. 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 beyond 200 °C [35,36]; 2) the strain rate range where DSA happens is lower than 1 s⁻¹, which is located in (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 [19,32,37], 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. 5–7), 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. 3c). According to the model proposed by Olson and Cohen [32], the fraction of the fraction of α' -martensite ($f^{\alpha'}$) at plastic strain (ε) is empirically described as the following formula:

$$f^{\alpha'} = 1 - \exp\{-A[1 - \exp(-\alpha\varepsilon)]^n\} \quad (7)$$

$$A = \bar{v}^{\alpha'} K p / (\bar{v}^{sb})^n \quad (8)$$

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 determined by chemical composition and temperature. Several investigations [19,32,37] 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 temperature above this value. However, large amount of α' -martensite is observed indeed in this work (Fig. 6). It can be hypothesized that the biaxial stress state promoted the $\gamma \rightarrow \alpha'$ transformation process. Hecker *et al.* [19,22] analyzed their experimental data using the transformation kinetics law proposed by Olson and Cohen [32], 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, Yeddu *et al.* [38] 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 joint promotion effect of decreasing SFE and biaxial stress on martensitic transformation compensated the impediment caused by adiabatic heating.

Twinning is another mechanism to accommodate additional strain in 304 SS by facilitating dislocation gliding through redirecting the grain orientation. Extensive mechanical twins were identified by EBSD and TEM analyses after biaxial deformation, as shown in Fig. 7a, 8a, 10d, and 11, indicating the substantial contribution provided by mechanical twins on strain hardening and energy absorption. As the occurrence of deformation twinning is directly related to the SFE of materials, which strongly depends on temperature, mechanical twinning activity is also influenced by temperature. By employing both simulation and experimental methods, Dávid Molnár *et al.* [34] concluded that the disappearance of deformation twins in 316L stainless steel at elevated temperatures was resulted from the increase in SFE and critical twinning stress. Their experiments were carried out under uniaxial tension and static strain rate ($5 \times 10^{-4} \text{ s}^{-1}$), revealing the gradual disappearance of deformation twins. The deterioration effect of increasing temperature on mechanical twinning can also be found on other investigations, such as [2,39]. In the present study, a rough comparison between the maps in Fig. 5 may demonstrate that the number of deformation twins kept constant with increasing plastic strain, even though the quantitative data of twins was not provided because of their irregular size.

Therefore, it should be sound to attribute the high plastic strain more to SIMT and dislocation glide instead of twinning.

Dislocation motion is the basic deformation mode in 304 SS and its dissociation into partials is the onset of α' -martensite embryos formation [40]. In the present study, large amount of dislocation with cell shape was found after biaxial stress deformation (Fig. 8b). 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 [41]. For instance, the dislocation motion may even transit from thermally activated slip to viscous drag mechanism with increasing strain rate [41,42]. Experimental study on the (111) $\langle 1\bar{1}0 \rangle$ type slip of aluminum single crystals at high strain rates (10^3 – 10^4 s $^{-1}$) under pure shear stress indicated the presence of viscous drag mechanism at high strain rate deformation [42]. 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 [43] and in oxygen-free copper [44], respectively. The investigation on copper via TEM characterization revealed that the dislocation density decreased with rising temperature at static uniaxial tensile testing [44]. Furthermore, the increased dislocation density may cause temperature rise during deformation. The investigation on Fe-3Si steel done by A. Eisenlohr *et al.* [45] revealed that the sudden temperature jump by 1 K within 0.04 s was induced by the increase in dislocation density. Unfortunately, scarce study of the effect of stress axiality 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 axiality and adiabatic heating.

4.2. 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. 6. However, in the Olson-Cohen model [32], $\gamma \rightarrow \alpha'$ 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 [19,37], while under dynamic condition, the content of martensite exhibits no such effect from available literature [19,46]. 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. 6), 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. 5, but their statistical data is not available in this study. Because currently there is 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.

5. Conclusions

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 biaxial loading.
2. The decreasing stacking fault energy (SFE) with temperature along with 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 $\sim 36\%$) 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 have the form of nanotwin bundles with a width of $\sim 5\text{ nm}$ to $\sim 20\text{ nm}$, after dynamic biaxial tensile deformation. Their inhomogeneous distribution in the microstructure and nanosize handicapped precise quantitative analysis.

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