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The significance of phase reversion-induced nanograined/ultrafine-grained structure on the load-controlled deformation response and related mechanism in copper-bearing austenitic stainless steel

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Abstract

The ingenious concept of phase reversion annealing involving cold deformation of parent austenite to strain-induced martensite, followed by annealing was used to obtain nano-grained/ultrafine-grained (NG/UFG) structure in a Cu-bearing biomedical austenitic stainless steel resulting in high strength-high ductility combination. Having employed the concept effectively, the primary objective of this study is to critically analyze the interplay between the load-controlled deformation response, strain-rate sensitivity and deformation mechanism of NG/UFG austenitic stainless steel via nanoscale deformation experiments and compare with its coarse-grained (CG) counterpart. The study demonstrated that the strain-rate sensitivity of NG/UFG was ~1.5 times that of the CG structure. Post-mortem electron microscopy of plastic zone surrounding the indents indicated that the active deformation mechanism was nanoscale twinning with typical characteristics of a network of intersecting twins in the NG/UFG structure, while strain-induced martensite transformation was the effective deformation mechanism for the CG structure. The fracture morphology was also different for the two steels, essentially ductile in nature, and was characterized by striations marking the line-up of voids in NG/UFG steel and microvoid coalescence in CG counterpart. The differences in deformation mechanisms between the NG/UFG and CG structure are attributed to the austenite stability – strain energy relationship. Furthermore, the presence of ~3 wt. % Cu in austenitic stainless steel had somewhat moderate effect on strain-rate sensitivity and activation volume at similar level of grain size in its Cu-free counterpart. Specifically, in the NG/UFG structure, the nanoscale twin density was noticeably higher in Cu-bearing austenitic stainless steel as compared to Cu-free counterpart, as Cu is known to increase the stacking fault energy.

Key words Austenitic biomedical alloy; Grain size; Cu addition; Activation volume.

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1. Introduction

Among stainless steels, austenitic stainless steels occupy about 70% of the market share in terms of applications. In view of their high corrosion resistance and relatively high ductility and toughness, austenitic stainless steels are widely used in various fields that include biomedical devices, marine engineering, nuclear industry and low temperature applications, to list a few [1,2]. However, their relatively lower strength restricts their application in structural and biomedical fields, which require relatively higher yield strength, besides high flexural and torsional strengths as well as wear resistance [3]. In order to expand the application field of austenitic stainless steels, a number of studies were aimed to develop advanced high strength, high ductility austenitic stainless steels for lightweight applications [4-6]. Grain refinement in essence provides a viable solution to enhance the strength of engineering metals and alloys [7, 8]. The concept of phase reversion annealing [9-18], involving cold deformation of metastable austenite to generate strain-induced martensite, was followed by appropriate temperature-time annealing sequence in order to obtain the desired nanograined or ultrafine-grained austenitic stainless steels.

In course of comparing the deformation behavior of traditional coarse-grained (CG) metals with nano-grained/ultrafine-grained (NG/UFG) metals, it has been previously suggested that the major mode for the ductile CG metals to plastically deform is controlled by the coupled activity of dislocations inside the grains (e.g. formation of dislocation pile-ups and cells), while, in the NG/UFG metals, the emission of partial dislocations from grain boundaries governed the plastic deformation process [5, 19]. Thus, it is expected that with significant decrease of grain size and

increase of yield strength, there should be a substantial change in deformation mechanisms.

In recent years, studies have indicated that NG/UFG metals are relatively more sensitive to strain rate, and the activation volume, which guides the rate-controlling mechanisms during the plastic deformation process [4, 20-26]. It was proposed that the presence of twins in UFG metals may effectively enhance strain-rate sensitivity and decrease the activation volume [20, 21].

The high ductility achieved in conventional CG austenitic stainless steels is governed by the gradual transformation of austenite-to-martensite, which increases the strain-hardening rate and postpones the commencement of localized necking [27-29]. However, the grain size of austenite is envisaged to play an important role in influencing the mechanical stability of austenite and delay phase transformation.

There is currently a significant interest in different grades of austenitic stainless steels (304, 316, 317 grades) containing copper from the perspective of promoting the antibacterial property of austenitic stainless steels [30-33]. However, systematic studies comparing the deformation behavior and strain-rate sensitivity of NG/UFG structure vis-à-vis CG structure, for instance, in type 304 steel, are rare. It is noteworthy that the deformation mechanism, particularly at low strain rate, is of particular significance for biomedical devices, because during micro/nanoscale deformation, when the bone rubs against the implant may induce twinning and/or strain-induced martensitic transformation, impacting the biological response of austenitic stainless steels [34].

The objective of the study described here is to elucidate the load-controlled deformation response and strain-rate sensitivity of NG/UFG and CG copper-bearing austenitic stainless steel and also compare the behavior with Cu-free steel. The experiments were conducted using a

nanoindenter, where the tested volume is comparable to the microstructure to the NG structure [21, 23] and the experimental conditions are identical. Nanoindentation technique was beneficial for studying micro/nanoscale deformation behavior of austenitic stainless steels and transformation-induced plasticity (TRIP) steels [35-39].

2. Materials and experimental procedure

The chemical composition of experimental austenitic stainless steel containing Cu (3.15 wt.%) is listed in **Table 1**. The steel was made in-house in a laboratory using standard melting practice. For cold rolling, the steel was received in the form of a hot rolled sheet, about 3 mm in thickness. The as-received steel sheet was cold rolled in a laboratory rolling mill to 1 mm thickness (66.7% reduction) and subsequently annealed at 800 °C for 10 s to obtain NG/UFG structure and 950 °C for 100 s to obtain the CG counterpart. The annealing was carried out in a Gleeble 3800 thermo-mechanical simulator. At 950 °C for 100 s, the final grain size of the experimental steel was similar to the as-received steel. The microstructure of NG/UFG and CG steel in terms of grain size was examined by transmission electron microscopy (TEM) and light optical microscopy, respectively. The annealed steels were subsequently tensile tested according to the ASTM standard E8. The fracture surface after the tensile tests was studied by scanning electron microscopy (SEM).

Two types of nanoscale deformation experiments were conducted. The first type was conducted in load-controlled mode at a loading rate of $2 \mu\text{Ns}^{-1}$ with the maximum load set to 0.5 mN. Here the objective was to observe any differences in load-displacement plots that may provide an insight on the deformation mechanism. The second type of experiment was conducted

in displacement-controlled mode, which involved indentation at various constant strain rates in the range $0.01 - 1 \text{ s}^{-1}$. The maximum displacement was fixed at 500 nm. Here the aim was to study the strain-rate sensitivity at low strain rate and compare it with that of Cu-free steel. The nanoindentation test system (Keysight Nanoindenter G200) consisted of a Berkovich three-sided pyramidal diamond indenter with a nominal angle of 65.3° and indenter tip diameter of 20 nm. An array of indents (10×10) were made with the indent gap of 10 μm . Post-mortem TEM study of indented NG/UFG and CG samples was carried out to explore the deformation mechanisms in the plastic zone surrounding the indented region. This involved removal of indented 3 mm punched disks from the mount and electropolishing from the side opposite to the indented surface, whereas the side with the indentations was masked with an aluminum foil. Using this approach, the area surrounding the indents present around the jet-polished hole, was electron transparent thus enabling study of the deformation behavior by TEM. During TEM studies, the focus was in the center of the deformation zone. The data presented here had excellent reproducibility, as confirmed by a number of experiments for each set of conditions.

3. Results

3.1 Microstructure of CG and NG/UFG austenitic stainless steels

Fig. 1 illustrates light and TEM micrographs of CG and NG/UFG structure, respectively. The average grain size of CG steel (cold rolled to 66.7% reduction and reversion annealed at 950°C for 100 s) was $22 \pm 5 \mu\text{m}$, while that of NG/UFG steel (cold rolled to 66.7% reduction and reversion annealed at 800°C for 10 s) mainly consisted of nanograins of size less than 100 nm and a few ultrafine grains of size $\sim 100\text{-}500 \text{ nm}$.

3.2 Mechanical properties

Tensile stress-strain plot depicting yield strength and elongation of CG and NG/UFG steels are presented in **Fig. 2**. The yield strength and elongation for CG steel are 297 MPa and 68%, respectively, while for the NG/UFG steel are 769 MPa and 38%, respectively. NG/UFG steel has shown ~2.5 times higher yield strength than CG steel at high level of elongation of 40%.

3.3 The tensile fracture surface

The fracture surface for NG/UFG and CG structures are presented in **Fig. 3**. In NG/UFG austenitic stainless steel, fine striations similar to those observed in fatigue fracture were observed, except that there is a line-up of voids along the striations. The striations on NG/UFG steel appear distinctly clear following processing the SEM micrographs with Image Pro software. It appears that tearing occurred along the striations. In the CG steel, microvoid coalescence leading to cup-and-cone type fracture was observed, which is commonly observed in ductile metals and alloys. Interestingly, the microvoids in CG austenitic steel are similar to the line-up of voids observed in NG/UFG steel along the striations. The microvoids corresponding to the coalescence in CG steel were only slightly larger in size in comparison to the line-up of voids along the striations in NG/UFG steel. The difference in the behavior of fracture surface is discussed in section 4.3.

3.4 Nanoindentation experiments

3.4.1 Load-controlled nanoindentation experiments:

Load-controlled nanoindentation experiments at a constant loading rate can elucidate the indentation-induced deformation phenomenon as a function of displacement (or strain) that is

difficult to achieve from the strain rate-controlled experiments. This is because the minimum strain rate available with the instrument is relatively quite high, and any discrete bursts in the load-displacement plots associated with dislocation nucleation or phase transformation cannot be recorded. **Fig. 4** presents representative load–displacement plots at constant loading rate of $2 \mu\text{Ns}^{-1}$. It may be noted that if the indentation hardness is assumed to be directly related to the strength, the loading rate can then be regarded to be equivalent to the indentation strain rate.

At loading rate of $2 \mu\text{N s}^{-1}$, there was no discontinuity in any of the load-displacement plots recorded for NG/UFG steel. A representative example is presented in **Fig. 4a**. On the contrary, the CG steel showed two discontinuous horizontal displacement bursts or arrests (referred as pop-ins) with the first one at a displacement of $\sim 20 \text{ nm}$ and at an applied load of 0.05 mN . The first pop-in in CG steel corresponds to dislocation nucleation [40, 41] and the subsequent pop-in in CG steel results from the strain-induced transformation of austenite-to-martensite (see section 4.2). The horizontal arrest represents the geometric softening caused by martensite variant selection, thereby minimizing the total energy change during austenite-to-martensite transformation process [36]. The initial region of NG/UFG steel and the region prior to the first pop-in in CG steel follows the power law relationship ($P \propto h^{1.5}$) and is in line with the Hertzian contact solution.

The similar penetration depth in nanoindentation experiments has been previously observed when the differences in yield strength are approximately two times. This is attributed to the indentation effect (IE) after 40-50 micrometers displacement for both the coarse-grained (CG) and nanograined/ultrafine-grained (NG/UFG) materials. The IE is expected to be less in NG steel

as compared to CG steel, which depends on the microstructural aspects such as grain size and dislocation density present within the material [42-45]. The size effects have been attributed to geometrically necessary dislocations that are introduced by strain gradients [46] and have recently been discussed for coarse-grained (CG) and ultrafine-fine grained (UFG) materials [47].

3.4.2 Strain rate controlled nanoindentation experiments

In addition to load-controlled nanoindentation experiments, CG and NG/UFG steels were also subjected to depth-sensing nanoindentation experiments at strain rates in the range 0.01 - 1 s^{-1} (0.01 , 0.1 , 0.5 , and 1 s^{-1}) to study the strain-rate sensitivity. The indentation strain rate is derived using the following equation [48]:

$$\dot{\epsilon} = \left(\frac{1}{h}\right) \frac{dh}{dt} \quad (1)$$

where, $\dot{\epsilon}$ is the indentation strain rate, h is the displacement, t is the loading time, dh/dt is the displacement rate. Here the displacement rate depends on the maximum displacement depth (set as 500 nm) and the required loading time. The data presented is an average of at least 10 experimental measurements with 95% confidence interval.

The hardness data of the samples that is directly obtained from the nanoindentation experiments was utilized to determine the strain-rate sensitivity. Hardness vs. strain rate plots for both the samples are presented in **Fig. 5** at various strain rates. It can be seen that hardness increased with increase in strain rate and was greater for NG/UFG steel in comparison to that of the CG steel at an identical strain rate. Please note that the unit of hardness is GPa, and hence there is an appreciable difference in the hardness values of NG/UFG and CG structures at a given strain rate.

3.5 Deformation structure

The results of post-mortem electron microscopy study of nanoindented samples are presented in **Figs. 6 and 7** for CG and NG/UFG steels, respectively. In the CG structure, only strain-induced martensite was observed (**Fig. 6**). In comparison, a number of representative electron micrographs are presented for NG/UFG structure, because this essentially constitutes the focus of the present study. Referring to the NG/UFG austenitic stainless steel, in general, a number of intersecting nanoscale twins were present in a number of regions (**Fig. 7a**). Furthermore, there were regions where high dislocation density was observed in the vicinity of nanoscale twins and the twin boundaries appeared extremely blurred because of the significant dislocation pile-ups (**Figs. 7b-d**). Thus, we can conclude that there was a clear and distinct transition in the mechanism of deformation from CG to the NG/UFG structure.

4. Discussion

4.1 Strain-rate sensitivity and activation volume

It is evident from **Fig. 5** that the hardness of NG/UFG steel was higher than that of CG steel. For instance, the average indentation hardness of CG and NG/UFG steel at the lowest strain rate (0.01 s^{-1}) was 0.9 GPa and 1.1 GPa, respectively.

The strain-rate sensitivity is calculated by using the following equation [20, 21]:

$$m = \sqrt{3}kT/v\sigma = 3\sqrt{3}kT/vH \quad (2)$$

where, m is a non-dimensional strain rate sensitivity index, k is the Boltzmann constant, T is the absolute temperature, σ is the flow stress, H is the hardness (which is generally assumed to be three times the flow stress) and v is the activation volume, which is the rate of decrease of the

activation enthalpy with respect to the flow stress at a fixed temperature [20, 21]:

$$v = \sqrt{3}kT\left(\frac{\partial \ln \dot{\epsilon}}{\partial \sigma}\right) \quad (3)$$

Strain-rate sensitivity parameter m and activation volume v provide insight into the sensitivity of flow stress to strain rate, and point out the similarity or difference in the deformation mechanism between NG/UFG and CG structures. According to the data in **Fig. 5**, the estimated strain-rate sensitivity values are 0.14 and 0.21 for the CG and NG/UFG structures, respectively, suggesting that the strain-rate sensitivity (m) of NG/UFG steel is 1.5 times of the CG counterpart. If we consider grain size based on a previous study [20], the strain-rate sensitivity m was only ~ 0.022 at a grain size of 100 nm and dropped further to ~ 0.012 at a grain size of 1000 nm for Cu and Ni. Thus, the m values of our experimental steel, both for CG and NG/UFG structures, are comparatively very high, suggesting that the effect of grain size is very small. According to the definitions of strain-rate sensitivity and activation volume stated in Eq. (3), activation volume (v) for both the CG and NG/UFG steels was calculated using **Fig. 5**. In order to simplify the expression, Burgers vector (b) was used. The activation volume of CG steel is $\sim 13b^3$, and the corresponding value for NG/UFG steel is $\sim 3b^3$. Although the activation volume value of CG is ~ 4 times larger than the NG/UFG structure, the difference is not very large for our stainless steel in absolute terms. These estimates point to the fact that the effective contribution of deformation mechanism to strain-hardening behavior in NG/UFG and CG structures is quite similar. Nevertheless, it is obvious that the indentation response of NG/UFG stainless steel to strain-rate sensitivity is greater than that of the CG material. Therefore, the strain-rate sensitivity of NG/UFG stainless steel is larger than that of CG steel. All the same, the differences in

strain-rate sensitivity and activation volume values of CG and NG/UFG structures are quite obvious and must be related to differences in their deformation mechanisms.

4.2 Deformation mechanism in NG/UFG and CG structures

Based on the results in **Figs. 6 and 7**, it is concluded that nanoscale twinning is an active deformation mechanism in NG/UFG steel, whereas strain-induced martensite formation is the effective deformation mechanism in CG steel. Both mechanisms, however, are responsible for the high ductility of the steel [49-51]. We know that twin nucleation is promoted by emitted multiple partial dislocations without dislocation rearrangement. In these situations, dissociation produces a fixed part and a twinning part; twin growth includes the twinning part experiencing double cross-slip [52]. At the same time, **Figs. 7b-d** implied that the existing twin boundaries (TBs) behaved similar to grain boundaries in acting as obstacles to strain propagation [53, 54]. The strengthening effect of twin boundaries acting as strong barriers to dislocation motion has also been demonstrated in an *in situ* TEM observation of the deformation process in a nanocrystalline Cu specimen [55]. Considering **Fig. 7d**, interestingly, the thickness of twin at the right top is evidently less than that at the left bottom. This can be attributed to the interaction between slip dislocations and the twin boundary. A perfect dislocation in the matrix (a primary plane) can dissociate into Frank sessile dislocations and Shockley partials, stopped by an obstacle such as twin boundary [54, 56]:

$$[\bar{1}01] \rightarrow \frac{1}{3} [\bar{1}\bar{1}1] + \frac{1}{6} [\bar{1}21] \quad (4)$$

The partial dislocation glides on the conjugate twinning plane, while the sessile dislocation is stopped at the intersection of the primary and conjugate planes. The pronounced dislocation

accumulation at the TBs leads to multiple consecutive interaction events between dislocations and the TB, which consequently decreases the thickness of twin lamellae [51] as marked in **Fig. 7d**. Therefore, twinning is an effective method to improve the strength and ductility of metallic materials with remarkable strain-hardening ability.

As regards the CG structure, the orientation of martensite transformed from austenite was determined using the electron diffraction pattern (**Fig. 6**). The orientation relationship between austenite and indentation-induced martensite followed the Kurdjumov-Sachs (K-S) orientation relationship, i.e., $\{111\}<110>\gamma//\{011\}<111>\alpha'$. Considering that each of the 24 K-S variants has a compression axis and two tensile axes for martensite transformation, termed as Bain distortion, the variants whose compression axis is almost parallel to the indentation direction have a high probability of selection during nanoindentation [36].

It is worth noting that TEM of all the NG/UFG and CG samples showed the above observations, while the areas far from the deformation zone did not exhibit the aforementioned observations.

Figs. 7 illustrates that mechanical twinning was an effective active deformation mechanism in NG/UFG structure. On the contrary, **Fig. 6** illustrates that strain-induced α' -martensite was an active deformation mechanism in the CG structure. Both mechanisms have positive effect on preventing strain localization and hence, improve the ductility. Therefore, twinning replaced the nucleation of strain-induced martensite, when the grain size was substantially reduced from CG to NG/UFG. This certainly is a consequential effect of the grain size refinement, thus leading to a noticeable increase in hardness and strength and presumably enhanced the austenite stability too

with decrease in grain size. Therefore, twinning becomes the preferred mechanism when the weighted average grain size is ~340 nanometers. Twinning promoted good ductility in "high strength" NG/UFG steel, but for the "low strength" CG steel, strain-induced martensite contributed to the high ductility. It is emphasized that twinning is a main factor leading to the high ductility of "high strength" NG/UFG structure and is an active and governing deformation mechanism, while for the "low strength" CG structure, the ductility expectedly was also very high, but without the occurrence of twinning.

Both deformation twinning and strain-induced α' -martensite formation are essentially strain hardening mechanisms that inhibit local strain and contribute to ductility. In addition, both mechanisms involve diffusionless shear of a constrained plate-like region of parent crystals. Twinning must be related to the enhanced contribution of grain boundaries that increase the stability of NG/UFG austenite, which limits the occurrence of strain-induced martensite, both of which effectively control the deformation mechanism and ultimately lead to fracture [57, 58]. It is generally believed that when fcc austenite is transformed into bcc martensite, anisotropic strain is introduced into the adjacent untransformed austenite to reduce the total strain energy [8, 59]. However, when the austenite grain size is smaller than the martensite lath, such as in NG/UFG structure, the number of martensite variants participating in an austenite grain is significantly reduced because of the high strain energy ($\sim 850 \text{ MJ/m}^3$), thereby reducing the ability to potentially nucleate the martensite [60].

4.3 Fracture behavior of NG/UFG and CG

There are interesting differences in the fracture mode of NG/UFG and CG steels (**Fig. 3**).

In NG/UFG steel, where nanoscale twinning was obtained, the striations with line-up of voids are observed (**Figs. 3c and d**). While in CG steel, when parent austenite transforms into martensite, the fracture is characterized by microvoid coalescence or dimple fracture (**Figs. 3a and b**).

While this aspect is currently being further studied via fracture toughness tests, the difference in fracture surface between the two steels merits a preliminary interpretation. We currently envisage that the fracture process in NG/UFG is step-wise or quasi-static in nature that produces a striated fracture. Striations had a spacing of $\sim 5 \mu\text{m}$. It is likely that the voids grow in front of the asserted crack. When the crack advances, the tearing of the intervoid area forms a ridge, which defines a new crack front. This process is repeated as a quasi-static crack growth process, such that a number of striations are observed.

4.4 The relationship between austenite stability and strain energy

The deformation mechanism in NG/UFG structure is related to the high density of grain boundaries, which led to the strength enhancement of NG/UFG austenite and prevented strain-induced martensite formation. This must be because of higher austenite stability with the decrease of grain size that led to the change in deformation mechanism.

Although the thermal stability of austenite is considered to be governed by grain size [58, 61, 62], the effect on mechanical stability is still not clear. As recently reported for TRIP steels [62], the stability of austenite grains is controlled by local carbon concentration. It is widely accepted that the transformation of austenite-to-martensite results in an anisotropic strain in adjacent untransformed austenite. The approximate equidistribution of transformation strain

demands that a number of multivariant transformations coincide in an austenite grain to minimize the total strain energy [58]. However, if the austenite grain size is equal to or smaller than the martensite lath, the possibility of several martensite laths appearing simultaneously in an austenite grain decreases with the decrease of space. Therefore, it is impossible to minimize the strain energy by martensitic transformation in NG/UFG steel. In summary, it is impossible to reduce strain energy when the NG/UFG austenite is transformed into martensite under single deformation mode due to space constraint effect. The following is a brief explanation on the effect of grain size.

Based on the physical energy and transformation from austenite-to-martensite [61], the mechanism of austenite stabilization induced by grain refinement is as follows. If austenite is transformed into martensite by single variant mode, the increase of elastic strain energy is defined by Eq. (5) [61]:

$$\Delta E_v = (1/2)E_1\varepsilon_1^2 + (1/2)E_2\varepsilon_2^2 + (1/2)E_3\varepsilon_3^2 \quad (5)$$

where E and ε are Young's modulus and elastic strain in each lattice plane, respectively. According to previous studies [60, 61], there are two methods of atomic motion during the fcc-bcc transformation lattice displacement: the first one is shear deformation of 36% to $[\bar{1}10]$ direction and the second one is anisotropic deformation accompanying the volume expansion of 4.5%, which includes 13.9% (ε_1) expansion to $[001]$ direction (1st direction), 7.0% (ε_2) contraction to $[\bar{1}10]$ direction (2nd direction) and 1.4% (ε_3) contraction to $[\bar{1}\bar{1}0]$ direction (3rd direction); then E_1 , E_2 and E_3 in the three directions are 132.1 GPa, 220.8 GPa and 220.8 GPa, respectively. According to Eq. (4), the evaluated increment of elastic strain energy is ~1840

MJ/m³.

By modifying Eq. (5), the increase of elastic strain energy can be obtained according to the following equation:

$$\Delta E_v = (1/2)E_1\varepsilon_1^2(x/d)^2 + \{(1/2)E_2\varepsilon_2^2 + (1/2)E_3\varepsilon_3^2\}(x/d) \quad (6)$$

where x is the thickness of the martensite plate and lattice strain is elastically accommodated over the space of austenitic grain (grain size: d). On substituting the value of Young's modulus and strain into Eq. (6), the increased elastic strain energy is:

$$\Delta E_v = 1276.1(x/d)^2 + 562.6(x/d) \quad (7)$$

For CG steel (average grain size of ~22 μ m), ΔE_v is ~6 MJ/m³ and for the NG/UFG steel (average size is ~340 nm), ΔE_v increases significantly to ~860 MJ/m³. Therefore, the nucleation ability of martensite reduces with finer grain size. Considering that multivariant transformation is difficult in the NG/UFG structure, the transformation from austenite to strain-induced martensite is inhibited. Therefore, the lattice displacement related to strain can be adjusted by dislocation sliding and twinning, suggesting that the twinning tendency increases with the decrease of grain size.

4.5 The effect of Cu addition on 304 stainless steel

We now discuss the effect of copper by comparing the results reported here with that of the Cu-free 304 stainless steel. The following are the differences between Cu-bearing and Cu-free 304 steels: the differences listed below are deduced from the work carried out by our group [63] in a manner identical to that described here. The strain-rate sensitivity for Cu-free steel (NG/UFG: 0.147; CG: 0.086) calculated by the nanoindentation experiments are not very

different from the values calculated here for both the CG structure and NG/UFG structure in Cu-bearing austenitic stainless steel (NG/UFG: 0.21; CG: 0.14). The similarity in activation volume of Cu-bearing (NG/UFG: $3b^3$; CG: $13b^3$) and Cu-free (NG/UFG: $6b^3$; CG: $19b^3$) is consistent with the deformation mechanism observed through the post-mortem electron microscopy of nanoindented Cu-bearing steel in this study and Cu-free NG/UFG and CG steels; i.e., twinning was observed in NG/UFG structure and strain-induced martensite in the CG structure. There was, however, an important difference in the degree of twinning (twin density) in NG/UFG structure of Cu-bearing (% area fraction: ~25%) and Cu-free (% area fraction: ~12%) 304 steels. The twin density was significantly greater in Cu-bearing 304 steel. The higher twin density in Cu-bearing steel is related to the fact that Cu promotes twinning because of its high stacking fault energy at 55 mJ/m^2 , which is well above the value facilitating martensitic transformation in steels ($<30 \text{ mJ/m}^2$)[64].

5. Conclusions

The load-controlled deformation response and strain-rate sensitivity of copper-bearing austenitic stainless steels (NG/UFG and CG) were studied using a nanoindenter (nanoscale deformation experiments) and post-mortem electron microscopy. The behavior was compared with that of the Cu-free austenitic stainless steel. The following are the conclusions:

- (1) The strain-rate sensitivity of NG/UFG structure was about 1.5 times (0.21) that of its CG counterpart (0.14). Using strain-rate sensitivity data, the activation volume of NG/UFG structure is about one-fourth ($3b^3$) of that of the CG structure ($13b^3$).
- (2) Post-mortem TEM studies indicated that the deformation mechanism of NG/UFG and CG

stainless steel was dramatically different. Deformation twinning resulted in high ductility of "high strength" NG/UFG steel, while in "low strength" CG steel, ductility was also very good but as a result of strain-induced martensitic transformation.

- (3) In NG/UFG structure, the twinning was the active deformation mechanism and the fracture morphology was characterized by striations (river markings) with line-ups of voids just along the striations. In contrast, in the CG structure, microvoid coalescence occurred leading to dimple type fracture with strain-induced martensite as the governing deformation mechanism.
- (4) The shift of deformation mechanism from strain-induced martensite in CG structure to nanoscale twinning in NG/UFG structure is related to the austenite stability that increased with the finer grain size.
- (5) The addition of Cu had moderate effect on the strain-rate sensitivity and activation volume of the austenitic stainless steel. However, there was noticeable difference in twin density, which was significantly greater in Cu-bearing steel compared to the Cu-free steel.

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Table 1. Chemical composition (wt.%) of experimental Cu-bearing austenitic stainless steel.

C	Si	Mn	Cr	Ni	Cu	S	P	Fe
0.023	0.55	0.85	17.40	7.32	3.15	0.011	0.025	Balance

Figure Captions

Fig. 1. (a) Light and (b) transmission electron micrographs of CG and NG/UFG structure, respectively in Cu-bearing austenitic stainless steel.

Fig. 2. Typical engineering stress-strain curves for CG and NG/UFG Cu-bearing austenitic stainless steels.

Fig. 3. SEM fractographs at identical magnifications illustrating microvoid coalescence type of fracture in CG (a and b) and line-up of voids along the striations in NG/UFG (c and d) in Cu-bearing austenitic stainless steels. Figures (b) and (d) are processed images with Image Pro software to clearly illustrate striations observed in NG/UFG Cu-bearing austenitic stainless steel (c).

Fig. 4. Load-displacement plots at fixed loading rate of $2 \mu\text{N s}^{-1}$ for NG/UFG and CG Cu-bearing austenitic stainless steels obtained via load controlled nanoindentation experiments.

Fig. 5. Hardness versus strain rate plots for CG and NG/UFG Cu-bearing stainless steels obtained via strain rate controlled nanoindentation experiments.

Please note that the hardness is in GPa. Thus, there is significant difference in the hardness of NG/UFG and CG Cu-bearing austenitic stainless steel.

Fig. 6. Post-mortem electron microscopy of the plastic zone surrounding the indented region in Cu-bearing CG austenitic stainless steel illustrates stain-induced martensite.

Fig. 7. Post-mortem electron microscopy of the plastic zone surrounding the indented region in Cu-bearing NG/UFG austenitic stainless steel.

Journal Pre-proof

Author Statement

C.Y. Hu conducted experiments under the guidance of R.D.K. Misra, M.C. Somani organized tensile data and contribution to the discussion, while C.G. Yang processed the steel and contributed to the discussion.

Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐ The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: