Mechanical properties of cryogenically-rolled type 321 metastable austenitic steel

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Abstract. In this work, the mechanical behaviour of cryogenically-rolled type 321 metastable austenitic steel was studied. For this purpose, the material was rolled at a temperature of liquid-nitrogen to degrees of rolling reduction, the evolved grain structure was characterized by the electron-backscatter diffraction (EBSD) technique and mechanical properties were evaluated by tensile tests. It has been shown that rolling to 10 pct. of thickness reduction resulted in the extensive strain-induced martensitic transformation thus giving rise to abrupt material strengthening as well as essential degradation of ductility. However, with a further increase in the rolling strain, the volume fraction of martensite tended to saturate thereby leading to stabilization of the mechanical characteristics.

1. Introduction
The possibility of a significant improvement of mechanical properties provides considerable interest in the development of ultrafine-grained (UFG) materials. Typically, this is achieved by applying severe plastic deformation methods [1], but these methods are laborious and difficult to use for manufacturing of commercial-sizeable products. However, in metastable austenitic steels, the UFG structure can be obtained by a combination of conventional cold rolling followed by annealing. This approach is based on the effect of the strain-induced martensitic transformation occurring during cold rolling and the subsequent austenite reversion occurring during appropriate heat treatment [2]. It is believed that the martensitic transformation essentially enhances the grain-refinement effect and an UFG structure can be produced at relatively low strain achievable during conventional rolling [3]. Moreover, lowering the deformation temperature down to the cryogenic range may additionally improve the grain-refinement efficiency. Specifically, it should (a) enlarge the driving force for both the strain-induced martensitic transformation and the thermal-assisted austenite reversion, and (b) stimulate mechanical twinning. In light of these advantages, the cryogenic processing of metastable austenitic steels has recently attracted considerable interest [4,5].
In the previous work, the effect of cryogenic rolling on the microstructure of type 321 metastable austenitic steel was studied in detail [5]. As expected, the imposed strain contributed to martensitic transformation which preferentially nucleated in the most-heavily strained areas, i.e. in the deformation bands. This, however, stagnated the normal development of the dislocation boundaries in austenite and thus retarded the grain refinement in this phase. The martensitic transformation was found to follow the well-known Kurdjumov-Sachs (K-S) orientation relationship, viz. \{111\}_\gamma//\{110\}_\alpha \text{ and } <110>\gamma//<111>\alpha$, and was characterized by a noticeable variant selection. The present work was undertaken to evaluate the mechanical properties of cryogenically-rolled material and elucidate the relationship between the microstructure and properties.

2. Material and Experimental Procedures

The program material comprised type 321 metastable austenitic stainless steel with chemical composition shown in table 1. The obtained material was rolled to 85% of hickness reduction at 950°C and then annealed at 1200 °C for 1 hour. This produced a fully recrystallized austenitic grain structure with a mean grain size of ~100 µm and a high fraction of annealing twins. This material condition is denoted throughout as the base material.

| Table 1. Chemical composition of type 321 metastable austenitic stainless steel, wt% |
|----------------|---|---|---|---|---|
| Fe | C | Cr | Ni | Ti | Si |
| balance | 0.12 | 18.6 | 10.2 | 0.7 | 0.76 |

The material was cryogenically rolled to 5, 10, 15, 23, 30 и 45 pct. of total thickness reduction. To ensure the conditions of cryogenic deformation, the rolling preform was soaked in liquid nitrogen and held for 15 minutes prior to rolling. Rolling was performed in a single pass, using a rolling speed of 160 mm/s in a cluster mill with working rolls with a diameter of 65-mm. The rolling stain to 45 pct. of reduction led to the significant adiabatic heating and concomitant loss of the cryogenic condition. Microstructural characterization of the cryogenically-rolled material was performed by electron backscatter diffraction (EBSD) and focused on the mid-thickness rolling plane. To provide reasonable statistics for measurements, a relatively large scan step size of 1 µm was used. The \(\alpha'\)-martensite was indexed as a body-centered-cubic phase. To eliminate spurious boundaries caused by orientation noise, the lower limit boundary-misorientation cut-off of 2° was employed. A 15° criterion was applied to differentiate low-angle boundaries (LABs) from high-angle boundaries (HABs). Mechanical behavior was evaluated using tensile tests. The tests were conducted at ambient temperature and a nominal strain rate of \(10^{-3} \text{ s}^{-1}\) using an Instron 5982 testing machine.

3. Results and discussion

The effect of cryogenic deformation on the grain structure is shown in figure 1, on the EBSD maps HABs are depicted as black solid lines. The key microstructural characteristics derived from the maps is summarized in table 2. Taking into account that a relatively coarse scan step size was used in the present study (1 µm), the measured martensite volume fraction, as well as the LAB fraction were most likely overestimated. Nevertheless, the obtained results are believed to qualitatively reflect real trends in the microstructure evolution.

The cryogenic rolling to 5 pct. of reduction provided only a minor effect on the grain structure, which was still dominated by relatively coarse austenite grains containing annealing twins (figure 1a). However, the increase in rolling reduction to 10 pct. resulted in principal changes. Specifically, the LAB fraction abruptly increased (table 2) thus evidencing a significant acceleration of the deformation-induced processes. Moreover, the evolved microstructure was characterized by a significant proportion of \(\alpha\) and \(\varepsilon\) martensite (figure 1b, table 2) thus indicating the strain-induced martensitic transformation. Two characteristic morphologies of \(\alpha\) martensite can be distinguished, i.e.,
(a) regular arrays of nearly parallel bands within austenite grains and (b) relatively coarse regions resembling the original grains. The first morphological type of martensite suggested the preferential development of phase transformation in the deformation bands (or mechanical twins), whereas the second morphological type may be interpreted as a plane-view of the \( \alpha' \)-transformed deformation bands.

Figure 1. Selected portions of EBSD grain-boundary maps showing grain structure and phase composition after cryo-rolling to (a) 5\%, (b) 15\%, (c) 30\%. The phase color code is shown in the bottom right corner of the figure.

Table 2. Phase composition, area fractions of HABs LABs, after cryo-rolling.

| Roll reduction | 5\%  | 10\% | 15\% | 23\% | 30\% |
|----------------|------|------|------|------|------|
| Austenit       | 0.998| 0.69 | 0.739| 0.723| 0.619|
| \( \alpha' \)-martensite | 0.001| 0.232| 0.205| 0.205| 0.354|
| \( \epsilon \)-martensite | 0.001| 0.078| 0.056| 0.072| 0.027|
| HABs           | 0.86 | 0.477| 0.462| 0.327| 0.309|
| LABs           | 0.14 | 0.518| 0.536| 0.669| 0.691|

A further increase in strain led to only minor changes in the microstructure morphology (figure 1c), the LAB fraction and the proportions of the martensitic phases (table 2), thus suggesting a stagnation in the microstructure evolution. Assuming the preferential nucleation of martensite in the deformation bands, as suggested above, the revealed saturation of its volume fraction may be explained in terms of depletion of the volume fraction of the deformation bands. After 30\% of accumulated strain, a subtle increase in the content of the \( \alpha' \)-martensite at due to the \( \epsilon \)-martensite is only worthy of remark (table 2). This may indicate a \( \epsilon \rightarrow \alpha' \) transformation.

Figure 2. Stress-strain curves of type 321 austenitic stainless steel with different cryo-rolling strains (a) and variation of reduction and elongation with cryo-rolling strain (b).
Tensile diagrams of the cryo-rolled material are summarized in figure 2 a. The effect of the rolling strain on the ductility and yield stress are shown in figures 2 b and 3 a, respectively. To quantify the effect of the rolling strain on the hardening efficiency, the strengthening rate was calculated as $h = \Delta \sigma / \Delta e$, where $\Delta \sigma$ is the increment in yield stress, and $\Delta e$ is the increase in rolling strain, the obtained results are shown in figure 3b.

![Figure 3](image)

**Figure 3.** Variation of yield stress (a) and strengthening rate (b) with cryo-rolling strain of type 321 austenitic stainless steel.

As expected, the rolling strain resulted not only in an essential degradation of ductility (figure 2), but also in a significant strengthening of the material (figures 2 a and 3 a). Remarkably, both effects tended to saturate after the rolling strain of 10 pct. (figures 2 b and 3 a). The saturation of the strengthening effect is also clearly seen in figure 3 b. It is evident that rolling to 10 pct. of strain provided the maximal strengthening effect, whereas a further increase in the rolling strain resulted in abrupt reduction of the hardening rate. All these observations are in excellent agreement with the evolution of the total martensite volume fraction (table 2) thus evidencing the dominant role of martensite in the microstructure-property relationship.

4. Summary
The results presented in this paper provide a strong evidence that rolling at cryogenic temperature promotes the strain-induced martensitic transformation, which preferentially nucleated in the deformation bands and resulted not only in a drastic (fourfold!) strengthening effect, but also in a dramatic degradation of ductility. It was found that the martensitic transformation began after the rolling strain to 10 pct. of thickness reduction, but the martensite volume fraction rapidly achieved a nearly saturation level and a further microstructure evolution was only characterized by the transformation of $\varepsilon$ martensite to $\alpha'$ martensite. The mechanical behavior broadly mirrored the evolution of the martensite volume fraction thus evidencing the dominant role of this phase in the microstructure-properties relationship.

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