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Abstract. The mechanical behavior of a chromium-nickel austenitic stainless steel with submicrocrystalline structures produced by multidirectional forging (MDF) to a total strain of ~ 4 at temperatures of 700 and 600°C was studied. This processing resulted in the formation of uniform ultrafine grained structure with an average crystallite size of 360 and 300 nm, respectively, and high dislocation density. The tensile tests were carried out in a wide temperature range 20-650°C. At ambient temperature, the yield stress (YS) comprised 900 MPa and 730 MPa in the samples subjected to MDF at 600 and 700°C, respectively. It should be noted that this strength was achieved along with elongations of 16% and 22% in the samples subjected to MDF at 600 and 700°C. The YS decreased and elongation-to-failure tends to increase with increasing test temperature and approaching 235 MPa and 51%, respectively, at 650°C. Effect of temperature on mechanical behavior of stainless steel with submicrocrystalline structure is discussed.

1. Introduction

Austenitic stainless steels are widely used for structural applications [1]. Such a widespread acceptance in various applications is associated with favorable combinations of their properties, i.e. ductility, toughness, formability, weldability, and corrosion resistance. However, engineering applications of Cr-Ni austenitic stainless steel are limited by a relatively low yield stress (YS) [2]. Extensive cold rolling provides an increase in YS from 230 to 710 MPa [2] due to dislocation strengthening, mainly. However, an 18-8 austenitic steel processed in high strength state invariably exhibits low ductility. More substantial increase in the strength of metals and alloys can be achieved by a structural strengthening through extensive grain refinement by severe plastic deformation [3-5]. Both dislocation strengthening and structural strengthening lead to a reduction in the ductility. However, extensive grain refinement provides ultimately better ductility than conventional cold-rolling. The aim of this work is to study the mechanical properties of a Super304H austenitic steel with ultrafine grained structure (UFG) produced by multidirectional forging (MDF). This steel is used for high temperature applications. It is know that the formation of sigma phase particles and $M_{23}C_6$ carbide on grain boundaries may significantly deteriorate ductility of austenitic steels [6]. Commonly, the M23C6 carbide and sigma phase particles precipitate at temperatures of 400-800°C in various chromium-nickel austenitic stainless steels. This study is particularly focused on the examination of the mechanical properties at temperatures ranging from 450 to 600°C to evaluate the effect of UFG structure on mechanical behavior at elevated temperatures.

2. Experimental Procedure

A Super304H austenitic stainless steel with chemical composition of 0.10%C–18.2%Cr–7.85%Ni–2.24%Cu–0.50%Nb–0.008%B–0.12%N–0.95%Mn–0.10%Si and the balance Fe (all in weight%) with an average grain size of about 7 μm was used as the starting material. Rectangular samples were subjected to MDF, which was carried out using isothermal multi-pass compression tests with a change in the compression

direction in 90° in order of three orthogonal axes from pass to pass at temperatures of 600 and 700°C. A total strain of 4 was applied for each sample.

Structural characterization was carried out on the samples sectioned parallel to the forging direction in the last pass by using an optical microscope, a Jeol JEM-2100 transmission electron microscope (TEM) and a Quanta 250 Nowa scanning electron microscope equipped with an electron back scattering diffraction (EBSD) analyser incorporating an orientation imaging microscopy (OIM) system. The grain sizes, fraction of high-angle and low angle boundaries were evaluated by OIM software (EDAX TSL ver.6). The boundaries with misorientation angles greater than 15 degrees were considered as high angle boundaries (HABs) and those with misorientations from 2 to 15 degrees were taken as low angle boundaries (LABs). The dislocation densities were evaluated by counting individual dislocations in the grain/subgrain interiors on at least eight arbitrarily selected typical TEM images for each data point.

The mechanical properties of processed samples were evaluated by means of tensile tests at temperatures from 20°C to 650°C by using flat specimens with gauge length of 12 mm and cross section of 3.0×1.5 mm. The tests were carried out by using an Instron universal testing machine (Model 5082) equipped with a three-zone split furnace. The flow curves were analyzed with a Bluehill software (Instron Corp.).

3. Result and Discussion

Typical deformation microstructures developed by MDF at temperatures of 600 and 700°C up to a total true strain of 4 are shown in Fig. 1 [7]. At 600°C, the average size of the true grains entirely delimited by high-angle boundaries is 300 nm (Fig. 1a). It is seen that these grains are subdivided by low-angle boundaries on subgrains. The deformation microstructure, therefore, consists of ultrafine (sub)grains bounded partly by LABs and partly by HABs, the portion of high-angle boundaries is 55%. The ultrafine (sub)grains involve high density of lattice dislocations of $\rho \sim 1.4 \times 10^{15}$ m^{-2}. In contrast, MDF at 700°C led to the formation of uniform UFG structure composing of nearly equiaxed ultrafine grains, which are entirely delimited by HABs (Fig. 1b). The average grain size in the sample processed at 700°C is 360 nm, the fraction of high-angle boundaries is 71%, and the lattice dislocation density is $\rho \sim 5 \times 10^{14}$ m^{-2}.
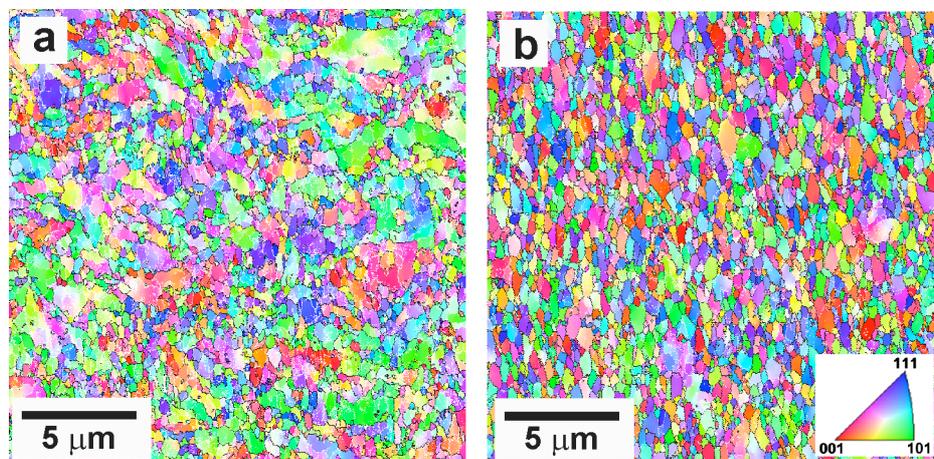

Figure 1. Typical OIMs of Super 304H-type steel after MDF (a) at 600°C, (b) at 700°C.

Low- and high-angle boundaries are indicated by white and black lines, respectively.

The engineering stress-strain curves obtained at different temperatures are presented in Fig. 2. It is seen that samples processed at 700°C show lower flow stress and significantly higher ductility than those process at 600°C. The shapes of σ-ε curves are nearly the same. At ambient temperature, a maximum stress is reached after prolonged strain hardening stage, and then the flow stress quickly decreases until fracture. Apparent steady-state flow could be distinguished. Increasing temperature to 500°C shifts the peak stress to lower strain and extends the stage of sharp softening. At T<600°C, increasing the temperature of the tensile tests leads to a decrease of the flow stress and ductility, concurrently. At T≥600°C, a maximum stress is reached after a very small strain and the stage of gradual strain softening after the peak stress tends to extend with increasing temperature. It is worth noting that the Super304H steel subjected to solution treatment shows extensive strain hardening in the temperature interval 500-650°C and jerky flow attributed to the pseudo Portevin-Le Chatelier (PLC) effect [8]. The total elongation increases with increasing temperature from 600 to 650°C up to the same level as at room temperature for the samples processed at 600°C. The samples processed at 700°C exhibit twofold increase in the total elongation with increase in temperature from 20 to 650°C. It is worth noting that the relatively high ductility at T≥600°C has no relation with extensive strain hardening, which should be necessary to provide necking resistance.
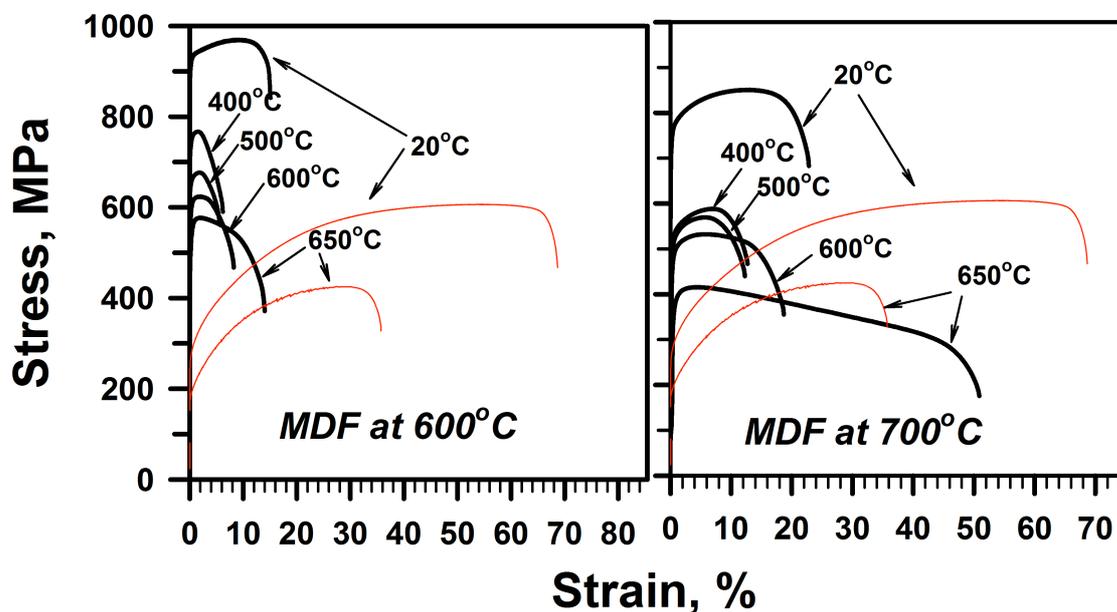

Figure 2. Tensile stress-strain curves of Super304H-type steel after MDF. The thin red lines correspond to the samples in heat treated condition.

The YS, the ultimate tensile strength (UTS), and the elongation-to-failure for the Super304H with UFG structure are summarized in Table 1. The mechanical properties of this steel after solution treatment [8] are presented for comparison. It is

worth noting that the average grain size of the solution treated steel was 18 μm, and the lattice dislocation density was $\rho \sim 7 \times 10^{12}$ m^{-2} [8]. It is seen that the YS increments are +220 and 160% for the steel processed at 600 and 700°C, respectively, due to extensive grain refinement and increased dislocation density. The corresponding increments of the UTS of +59 and 39% are significantly less, and ductility drops by a factor of ~4 and ~3, respectively. The increments in YS and UTS owing to MDF decrease with increase in the temperature of the tensile tests. At 650°C, the YS increments are +160 and 85% for the steel processed at 600 and 700°C, respectively (Table 1). At the same time, the UTS values for the Super304H steels in solution treated condition and subjected to MDF at 700°C are essentially the same, and elongation-to-failure is even higher by a factor of 1.5. The +36% increase in the UTS is retained for the steel subjected to MDF at 600°C. It is worth noting that the Super304H steel processed by MDF exhibit low values of ductility in the temperature interval 400-600°C, while the starting material show high elongation-to-failure and the pseudo PLC effect [8]. No any evidence of serrated flow was found for this steel processed in high strength state through MDF. Therefore, the suppression of the pseudo PLC effect is accompanied by a strong decrease in ductility.

Table 1. Mechanical properties of Super304H-type steel subjected to MDF at 600°C and 700°C and after solid solution treatment. * in .

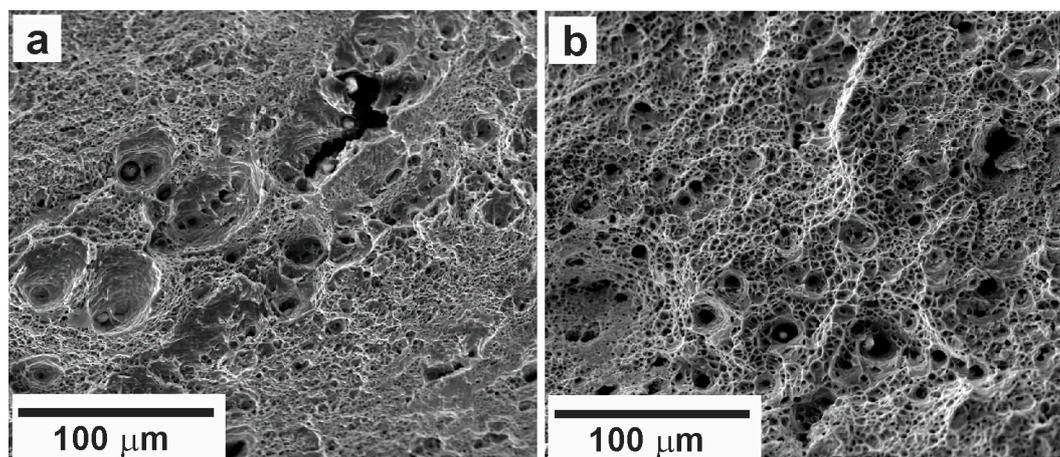|		20°C	400°C	500°C	600°C	650°C
YS, MPa	MDF 600°C	900	710	640	580	510
	MDF 700°C	730	485	470	460	350
	In heat treated condition	280	-	188	190	189
UTS, MPa	MDF 600°C	970	770	680	620	580
	MDF 700°C	850	590	570	530	415
	In heat treated condition	610	-	509[8]	450[8]	425
Elongation, %	MDF 600°C	16	7	6	8	14
	MDF 700°C	23	13	12	19	51
	In heat treated condition	65	-	43	41	34

Figure 3. Fracture surfaces of samples subjected to MDF at 600°C and then tensile tested at 500°C (a) and 650°C (b).

Typical SEM micrographs of the fractured surfaces of samples subjected to MDF at 600°C and then tensioned at 500°C and 650°C are shown in Fig. 3. The ductile fracture is in dominant [9]. Fracture surfaces exhibit well-defined various dimple sizes. Coarse dimples are nucleated by relatively coarse particles, while no particles were found on the bottom of fine dimples. It seems that numerous nucleating sites for microvoids are activated at grain, and the non-uniform distribution of boundary particles leads to growth of several isolated microvoids to a large size before coalescence [9]. At 500°C, a high non-uniformity in distribution of dimple sizes is observed (Fig. 3a). In addition, an evidence for intergranular dimple rupture can be found (Fig. 3a). At 650°C, the fracture consists of nearly uniform dimples (Fig. 3b). The dimensions of coarse dimples observed after tensile tests at 650°C are smaller than those at 500°C. Only intragranular rupture takes place.
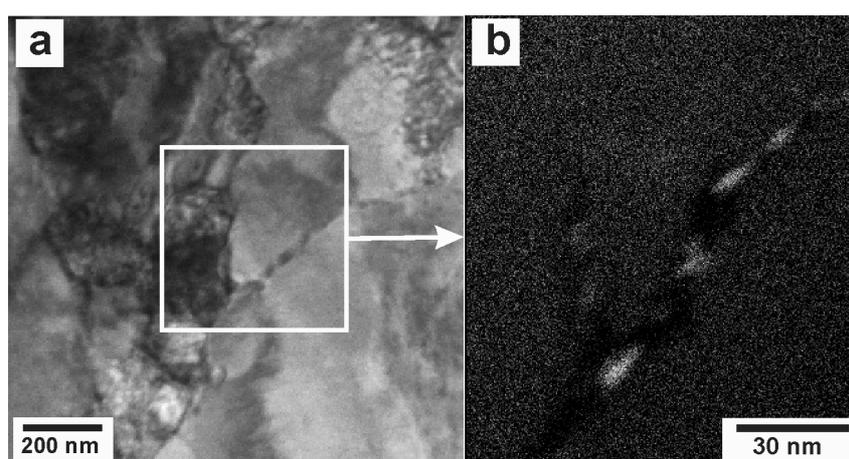

Figure 4. STEM micrograph (a) and Cr element mapping (b) of samples subjected to MDF at 600°C and then tensile tested at 500°C.

Scanning transmissions electron microscope (STEM) micrograph and the elemental mapping for chromium distribution are shown in Fig 4. It can be seen that the

distribution of Cr atoms is heterogeneous. The Cr atoms segregations appear along the grain boundaries that leads to precipitation of $M_{23}C_6$ carbides on these boundaries. The Cr-free zones are observed in vicinity of these carbides. These precipitations seem to initiate the intergranular fracture that leads to premature fracture in the temperature interval 400-600°C. It should be noted that the chromium segregations are not observed in the samples, which were tensile tested at 650C irrespective of the MDF temperature, and these samples exhibit high ductility.

Thus, the intense plastic straining by MDF is very effective in increase of the YS of austenitic stainless steel, which is caused by both the extensive grain refinement and the accumulation of high dislocation density. A decrease in temperature of MDF results in significant increase in strength.

4. Conclusions

The multidirectional forging of Super304H steel at temperatures of 600°C and 700°C leads to the formation of ultrafine grained structure with an average size of 300 and 360 nm, respectively, and correspondingly high densities of lattice dislocations of 5×10^{14} and 1.4×10^{15} m^{-2}. The multidirectional forging of Super304H steel provides threefold increase in yield stress in a wide temperature range from 20 to 650°C.

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