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To cite this article: V Torganchuk et al 2019 J. Phys.: Conf. Ser. 1270 012039

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IOP Conf. Series: Journal of Physics: Conf. Series **1270** (2019) 012039 doi:10.1088/1742-6596/1270/1/012039

Grain sizes and dislocation densities in fcc-metallic materials processed by warm to hot working

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Abstract. Our just received results on the deformation microstructures evolved in a medium-Mn austenitic steel during large strain warm to hot rolling were analyzed with a reference to other alloys with face centred cubic lattices and relatively low stacking fault energies. The structural changes were characterized by the development of dynamic recovery (DRV) and dynamic recrystallization (DRX). The deformation grain size decreased while the dislocation density increased with a decrease in deformation temperature. Both the grain size and the dislocation density could be expressed by power law functions of temperature-compensated strain rate, i.e., Zener-Hollomon parameter, with exponents of -0.33 and 0.2, respectively, in the case of discontinuous DRX. On the other hand, the exponents of -0.1 and 0.06 were obtained for the grain size and dislocation density dependencies on the temperaturecompensated strain rate, respectively, under conditions of warm deformation accompanied by DRV and continuous DRX. Therefore, a power law function could be obtained between the grain size and the dislocation density with a grain size exponent of -0.6, irrespective of the mechanisms of microstructure evolution in a wide range of deformation conditions.

1. Introduction

Structural steels and alloys are commonly subjected to various thermo-mechanical treatments, which result in required combinations of mechanical properties of semi-products owing to appropriate microstructures. The most important microstructural parameters affecting the mechanical properties of metallic materials are the phase content including the distribution of dispersed particles, the grain size, and the dislocation density [1-4]. The phase content depends on the alloying design and can be realized for improving the properties in multiphase systems, whereas a beneficial combination of the grain size and the dislocation density enhances mechanical properties of almost all structural steels and alloys. Therefore, studying the mechanisms of microstructure evolution in metals and alloys during thermo-mechanical processing is of great practical importance.

Warm to hot working of metallic materials is frequently accompanied by dynamic recrystallization (DRX) [5-7]. Two DRX mechanisms are discussed in relevant literature [6]. Those are discontinuous DRX and continuous one. The former takes place in metals and alloys with relatively low stacking fault energy (SFE) subjected to hot working. This is a classical DRX mechanism, which has been revealed several ten years ago and clarified in numerous studies [5]. In contrast, the mechanism of continuous DRX has not been well documented and it is still arousing some debates. It is currently agreed that continuous DRX may occur in metals and alloys with relatively high SFE under hot working conditions as well as in almost all metallic materials irrespective of SFE under conditions of warm or even cold working [7]. The DRX grain size (D) depends sensitively on the deformation condition, which can be adequately represented by temperature-compensated strain rate, i.e., Zener-

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Hollomon parameter, $Z = \dot{\varepsilon} \exp(Q/RT)$, where $\dot{\varepsilon}$ is the strain rate, Q is the activation energy, R and T are the universal gas constant and absolute temperature, respectively. A power law function, $D \sim Z^{-n}$, with an exponent of approx. 0.4 has been reported for discontinuous DRX during hot working of face centred cubic (fcc) materials with low SFE [8]. This strong temperature/strain rate dependence of the DRX grain size becomes much weaker as deformation temperature decreases leading to a change in DRX mechanism from discontinuous to continuous [8]. Correspondingly, an exponent in the power law function tends to decrease below 0.1. In contrast to the DRX grain size, the effect of deformation conditions and DRX mechanisms on the dislocation substructures in warm to hot worked steels and alloys has not been studied in detail. An inverse proportional relationship has been suggested between the DRX flow stress and the subgrain/cell size in DRX microstructures [1, 9]. However, the variation of dislocation density in DRX microstructures has not been explained satisfactorily. The aim of the present study, therefore, is to elaborate the relationship between the deformation conditions within the warm to hot working domain and the microstructural parameters, i.e., the deformation grain size and dislocation density, and their effect on the strength of a medium-Mn austenitic steel as a typical representative of fcc-metallic materials with low SFE.

2. Experimental

The medium-Mn steel, Fe-12%Mn-0.6%C-1.5%Al (all in wt.%) was produced in the Central Research Institute for Machine-Building Technology, Russia. The steel was characterised by a uniform austenite microstructure with an average grain size of 80 μ m that developed by hot rolling at 1423 K. The steel samples were rolled to a total reduction of 60% at different temperatures ranging from 773 K to 1373 K. The rolling reduction in each rolling pass was 10%. The samples were reheated to rolling temperature after each rolling pass. The structural investigation were carried out by an orientation imaging microscopy (OIM) based on electron backscattering electron diffraction (EBSD) analyser set at a Quanta Nova NanoSEM 450 scanning electron microscope (SEM). The mean grain size and the kernel average misorientation were evaluated using TSL OIM Analysis software, ver. 6.2. The grain size was evaluated by a linear intercept method along the normal direction (ND). The dislocation density was calculated as $\rho = 1.15 \,\theta_{KAM} / (b h)$, where θ_{KAM} is the kernel average misorientation, b is the Burgers vector, and h is the step size in the OIM map [10]. The tensile tests were carried out at ambient temperature using an Instron 5882 universal testing machine and dog-bone specimens cut along the rolling direction (RD).

3. Results and discussion

3.1. Deformation microstructures

The effect of rolling temperature on the deformation microstructures evolved in the steel samples is illustrated in figure 1. Warm rolling at 773 K to 973 K brings about the elongation of original grains along the rolling direction and the development of numerous microshear bands, which is accompanied by the formation of strain-induced grain boundaries (figures 1a and 1b). In contrast, the deformation microstructures evolved during hot rolling at 1073 K to 1373 K consist of almost equiaxed grains containing annealing twins (figures 1c and 1d). Such microstructures are typical of discontinues DRX that takes place in low SFE metals during hot working [5]. It can be concluded, therefore, that the studied range of rolling temperatures covers both the hot deformation domain of discontinuous DRX and warm deformation domain of dynamic recovery (DRV) or continuous DRX.

The transverse grain size (D) evolved in the present steel samples after rolling at different temperatures is shown in figure 2 as a function of temperature–compensated strain rate (Z) along with data from other studies on DRX microstructures [11-15]. The grain size decreases with a decrease in temperature and/or an increase in strain rate. Within the hot deformation domain with $Z < 12^{12} \text{ s}^{-1}$, the grain size exhibit a rather strong power law relationship with Z with an exponent of 0.33. On the other hand, a power law function with much smaller exponent of about 0.1 holds between the grain size and the deformation conditions, i.e., Z. This change in the relationship between D and Z has been

discussed in terms of DRX mechanisms [8, 14]. Namely, transition from discontinuous DRX to continuous one, when DRV serves as a softening controlling mechanism, with a decrease in deformation temperature occurs along with such a change in the DRX grain size dependence.



Figure 1. OIM micrographs of the deformation microstructures evolved in a medium-Mn steel processed by warm to hot rolling at 873 K (a), 973 K (b), 1073 K (c), 1173 K (d). The colours indicate the crystallographic direction along the normal direction (ND).

A reduction of the grain size with a decrease in deformation temperature is accompanied by an increase in the internal stresses within the warm to hot worked microstructures. The distribution of internal distortions can be mapped by the kernel average misorientation (figure 3). An average value of the kernel average misorientation increases from 0.5° to 2.9° as the rolling temperature decreases from 1373 K to 773 K. It should be noted that the deformation microstructures are characterized by inhomogeneous distribution of kernel average misorientations. The large kernel average misorientations are located near the grain boundaries and along the microshear bands in the warm rolled microstructures (figures 3a and 3b). The distribution of kernel average misorientations becomes more uniform in the discontinuous DRX microstructures, although different ultrafine grains evolved by hot rolling at 1073 K exhibit different levels of internal distortions (figure 3c). The non-uniform distribution of internal distortions in the ultrafine grained microstructures that develop during hot working is associated with discontinuous DRX mechanism, when the just nucleated stress-free DRX grains cyclically replace the previously grown work hardened grains. The kernel average misorientation decreases with an increase in rolling temperature; remarkable distortions can be observed only near the grain boundaries in the DRX microstructures evolved during hot rolling at a relatively high temperature (figure 3d). Such distribution of the internal distortions indicates that any post-DRX scarcely affected the deformation microstructures after the final rolling pass.

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Figure 2. Effect of deformation conditions on the deformation grain size in a medium-Mn steel and other fcc-alloys with low to medium SFE [11-15].



Figure 3. Kernel average misorientations in the deformation microstructures evolved in a medium-Mn steel processed by warm to hot rolling at 873 K (a), 973 K (b), 1073 K (c), 1173 K (d).

The effect of deformation conditions on the corresponding dislocation density is shown in figure 4. Some available date for the dislocation densities measured by direct transmission electron microscopy observations in fcc-metallic materials with low-to-medium SFE [12, 15, 16] are also represented in figure 4. It is clearly seen that the change in the DRX mechanism at Z of approx. 10^{12} s⁻¹ is accompanied by the change in the temperature/strain rate dependence of dislocation density. Namely, an exponent of about 0.2 in a power law function of $\rho \sim Z^q$ decreases to 0.06 as deformation conditions change from hot working accompanied by discontinuous DRX to warm working with continuous DRX at sufficiently large strains.



Figure 4. Effect of deformation conditions on the dislocation density in the deformation microstructures in a medium-Mn steel and other fcc-alloys with low to medium SFE [12, 15, 16].

3.2. Tensile properties

A series of the stress-strain curves obtained by tensile tests of the warm to hot rolled steel samples is shown in figure 5. In spite of the different microstructures evolved by warm or hot rolling, the steel samples demonstrate similar tensile behaviour. The strain hardening rapidly decreases at an early plastic deformation resulting in pronounced stage of uniform elongation, when the strain hardening slightly decreases with straining, followed by necking and rapid failure. Such tensile behaviour is typical of high-Mn austenitic steels exhibiting the effects of twinning/transformation induced plasticity [17]. A decrease in the rolling temperature significantly strengthens the steel samples. The yield strength increases from 340 MPa to 950 MPa as the rolling temperature decreases from 1373 K to 773 K. It should be noted that the strengthening is not accompanied by remarkable change in the strain hardening, although total elongation decreases.

3.3. Structure-property relationship

The results obtained in the present study and those reported in other papers for austenite and copper testify to unique power law relationships held between the deformation microstructures the deformation conditions. The most important that these relationships suggest a unique power law function between the deformation grain size and dislocation density. Indeed the power law functions of $D \sim Z^{-0.33}$ and $\rho \sim Z^{0.2}$ obtained for hot working conditions results in a power law function between ρ and D with a grain size exponent of -0.6. On the other hand, the grain size and dislocation density can be related to temperature-compensated strain rate with exponents of -0.1 and 0.06, respectively, in

the case of warm working. These dependencies result in the same power law relationship between the dislocation density and grain size with an exponent of -0.6. Figure 6 represents the variation of dislocation density with grain size for the present steel samples and other austenitic steels and copper alloys [12, 13, 15, 18] and confirms the statement above. The same relationship between the grain size and dislocation density has been reported for continuous DRX microstructures evolved in austenitic stainless steels during warm rolling to large total strains [15]. The linear plots in figure 6 suggest that a unique relationship between the grain size and dislocation density may held in DRX microstructures evolved in wide range of materials and deformation conditions irrespective of DRX mechanisms.



Figure 5. Tensile stress-strain curves for a medium-Mn steel subjected to warm to hot rolling at the indicated temperatures.



Figure 6. Relationship between the dislocation density and the grain size in a medium-Mn steel and other fcc alloys with low SFE subjected to warm to hot working [12, 13, 15, 18].

7th International Conference on Recrystallization and Grain Growth	IOP Publishing
IOP Conf. Series: Journal of Physics: Conf. Series 1270 (2019) 012039	doi:10.1088/1742-6596/1270/1/012039

The yield strength of work hardened metallic materials processed by large strain deformation can be expressed by a modified Hall-Petch-type relationship, $\sigma_{0,2} = \sigma_0 + k D^{-0.5} + \Delta \sigma(\rho)$, where σ_0 is the strength of the same dislocation-free material with infinite grain size, k is the grain size strengthening factor, and the last term represents the dislocation (substructural) strengthening [15, 19]. The latter is commonly calculated by Tailor-type equation, $\Delta\sigma(\rho) = \alpha Gb\sqrt{\rho}$, where α is a numerical factor, and G is the shear modulus. The revealed unique relationship between the deformation grain size and dislocation density, e.g., $\rho = 1.36E13 + 2.5E14 \text{ D}^{-0.6}$ for the present steel samples in figure 6, enables one to predict the strength of warm to hot worked materials by using either D or p. It should be noted that attempts to evaluate the strength by using either grain size or dislocation density have been discussed in other studies [15, 20, 21]. Setting $\alpha = 0.82$ [22], the Hall-Petch-type plot for the present steel samples is shown in figure 7a. Note here that the values of the first term of 172 MPa and the grain boundary strengthening factor of 648 MPa $\mu m^{0.5}$ in figure 7a are similar to those reported in other studies on the grain size strengthening [20, 22, 23]. The relationships between the experimental yield strength of the present steel samples and those calculated by using either grain size, $\sigma_{0,2}(D)$, or dislocation density $\sigma_{0,2}(\rho)$ as a unique variable are presented in figure 7b. A good correspondence between the experimental and both calculated values validates the presented treatment.



Figure 7. Grain size strengthening (a) and relationship between the experimental and calculated yield strengths (b) for a medium-Mn steel subjected to warm to hot rolling.

4. Summary

The warm to hot rolling of a medium-Mn austenitic steel at different temperatures of 773 K to 1373 K was accompanied by the development of dynamic recovery (DRV) and dynamic recrystallization (DRX). The latter readily developed at temperatures above 1073 K by the discontinuous DRX mechanism. The transverse grain size measured along the normal direction decreased with a decrease in deformation temperature and could be expressed by power law functions of temperature-compensated strain rate, i.e., Zener-Hollomon parameter (Z), with exponents of -0.33 in the range of Z < 10^{12} s⁻¹ corresponding to hot working accompanied by discontinuous DRX and -0.1in the range of Z > 10^{12} s⁻¹ corresponding to warm working with DRV as the main restoration process. The change in the dislocation density clearly correlated with that of the deformation grain size. Namely, the dislocation density increased with Z from power law with an exponent of 0.2 during hot working or 0.06 under warm working conditions. Correspondingly, a unique power law relationship with a grain size exponent of -0.6 was obtained between the deformation grain size and the dislocation density in a wide range of warm to hot rolling conditions, irrespective of the mechanisms of microstructure

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evolution. Therefore, the yield strength of the rolled steel samples could be expressed by a modified Hall-Petch-type relationship, which includes the dislocation strengthening, by using the grain size or dislocation density as a unique variable.

Acknowledgements

Authors acknowledge with gratitude the financial support received from the Ministry of Education and Science, Russia, under Grant No. 11.3719.2017/PCh (11.3719.2017/4.6).

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