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Effect of forming conditions on the softening behavior in coarse grained structures

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ABSTRACT

Polycrystalline nickel with a purity of 99.99 wt% and different starting grain sizes of 240 μm and 770 μm has been investigated. The effects of the initial grain size on the hot deformation behavior were studied by compression tests at various forming conditions. The microstructure was captured after deformation, using electron back scatter diffraction technique (EBSD). A variation of the initial grain size has unexpected effects on the acting softening mechanisms. A coarsening in the starting microstructure leads to a higher flow stress and a remarkable higher hardening at elevated temperatures. Furthermore, the nucleation of dynamic recrystallized grains is enormously retarded due to the less pronounced grain boundary bulging in a coarser grained microstructure compared to a finer one. As a consequence, discontinuous dynamic recrystallization generates a metastable grain size, that is finer than the steady state grain size.

1. Introduction

The refinement processes during warm forming of coarse grained structured metallic materials are of technical interest to improve the mechanical properties and to generate a well defined microstructure. Dynamic recrystallization during and after warm forming is the unique process that can be used to refine the microstructure of metals without phase transformations (Ni, Cu, Al, γ-iron). Dynamic as well as static recovery and recrystallization are the dominant processes controlling the final microstructure.

The main characteristics of dynamic recrystallization and recovery under different forming conditions have been analyzed by numerous authors, see for example [1–10]. In low to medium stacking fault energy materials, such as austenitic stainless steels, copper and nickel, discontinuous dynamic recrystallization (DDRX) is the main recrystallization mechanism. The microstructural evolution is characterized by the formation of new equiaxed grains at preexisting grain boundaries [11–15] or deformation heterogeneities which have a high local misorientation [16–18]. Furthermore, it has been shown that the forming conditions, temperature and strain rate have a pronounced effect on the transition from single to multiple peak behavior in the stress–strain response. In addition to the forming parameters, this transition in flow behavior is strongly influenced by the initial grain size and has been extensively investigated by Sakui et al., Sakai and Jonas [19,20]. These experimental observations clearly show that the shape of the flow curve is determined by the nucleation density of grains. Sakai et al. proposed in their model that grain boundary sliding and annealing twinning are the basic steps required for DRX nucleation [21,22]. Miura et al. pointed out the particular importance to previous grain-boundary serration and grain-boundary migration mechanisms, which can be accompanied with the twinning process [12,23]. In marked contrast to the clear relation between grain boundary mobility and nucleation of new grains, which has already been investigated by many authors, there are only a few publications on the influence of grain boundary mobility and grain size on the stress–strain response. In the work of Angella et al., performed on an austenitic stainless steel 316L, strong strain induced boundary migration (SIBM) events helped to trigger the nucleation process [24]. Additionally, they reported from an anomalous flow behavior, caused by SIBM, where a coarser grained material had a higher hardening response as a finer grained one.

The present work examines the influence of initial grain size on the flow behavior and microstructural evolution of coarse grained nickel. Compression tests at various forming conditions analyzed with the orientation imaging microscopy technique, serve as the basis for the understanding of the underlaying mechanism. In order to better understand the phenomena controlling the dependence of the structural evolution and flow stress during hot forming on

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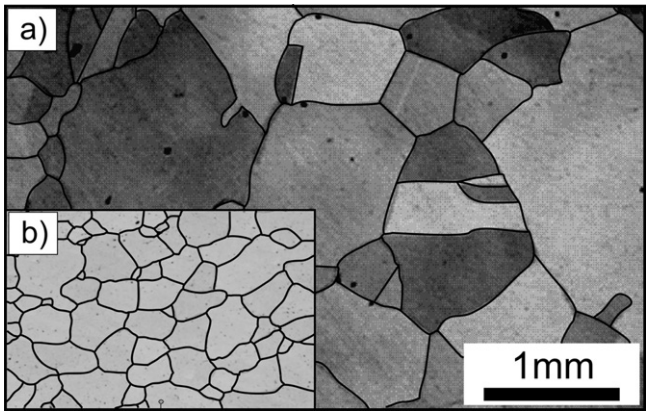

Fig. 1. Initial microstructure of nickel 45% cold worked and annealed at 1180 °C (a) for 48 h with D_0 = 770 μm and (b) for 1 h with D_0 = 240 μm.

the initial grain size a detailed analysis, using the EBSD technique is performed.

2. Experimental procedure

Pure nickel (99.99 wt%) polycrystalline samples were deformed by compression tests at different warm forming temperatures to investigate the influence of initial grain size on the structural refinement process. The compression test samples, with a diameter of 10 mm and a height of 12 mm, were machined from plates. The specimens were deformed in an displacement controlled mode with constant cross-head speeds of 5 mm/min or 50 mm/min, equivalent to an average strain rates of $\dot{\epsilon} \approx 0.01\ s^{-1}$ and $\dot{\epsilon} \approx 0.1\ s^{-1}$. A compression test unit "Schenck-Trebel" was used. Temperature was held constant at ambient temperature, 450 °C, 830 °C, 1000 °C or 1180 °C during the compression. To minimize static- and post-dynamic softening processes, the samples were water quenched after the deformation step within one second. Two sets of specimens having different initial grain sizes of D_0 = 240 μm and D_0 = 770 μm were used. The different microstructural features of the finer grained and the coarser grained sample are shown in Fig. 1. Both structures consist of equiaxed grains. One distinctive feature of the coarser grained material is the occurrence of few annealing twins.

$$\Delta g_k = \frac{1}{12}\sum_{i=1}^{12} \Delta g_i \tag{1}$$

The structural analysis of undeformed and deformed material is based on the measurements of the local crystal orientation. These measurements were carried out with an EBSD-SEM system (a TSL EBSD system interfaced to a LEO 1525). To get rid of any preparation artifacts the sample surfaces were polished mechanically as well as electro-chemically. The grain boundary profile and differences in crystallographic orientation of the samples after the forming process are plotted as Inverse Pole Figure (IPF) maps and Kernel Average Misorientation (KAM) maps. Fig. 2 represents the kernel used for misorientation calculation. In this mode, the misorientation between the center point and all points at the perimeter of the kernel are measured. The local misorientation value Δg_k assigned to the center point is the average of these misorientations Δg_i as given in equation 1[25]. The procedure is similar as in the analyses proposed by Vorhauer et al. [26]. The second neighbors at the perimeter were taken to weaken the influence of single erroneous data points on the results.

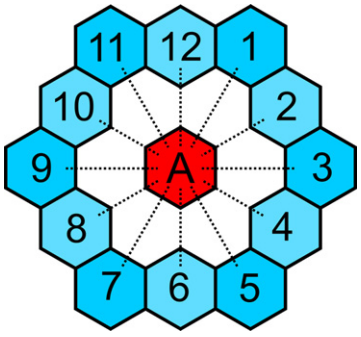

Fig. 2. Kernel based misorientation calculation.

3. Results

3.1. Stress–strain behavior

In Fig. 3(a), the stress–strain curves and in Fig. 3(b) the corresponding hardening curves of different initial microstructures are plotted. The samples were deformed at different temperatures with a constant crosshead speed equivalent to a strain rate of about $\dot{\epsilon} \approx 0.1\ s^{-1}$. Stress–strain curves for the finer grained samples are represented by dashed lines and those for the coarser grained specimens are visualized by solid lines. For deformation experiments at ambient temperature and 450 °C a plateau stress could not be reached due to the limitation in forming capacity. Several differences in the shape of the curves, when comparing the influence of initial grain size were, found. The deformation experiments at ambient temperature show a "typical" cold working behavior. In the forming temperature range from 450 °C to 1000 °C the coarser grained material has a higher flow-stress and a higher hardening than the finer grained material for strains larger than few percents. At a deformation temperature of 1180 °C this effect is retarded and does not become visible before the peak strain in the finer grained material was reached. Forming at elevated temperatures results in a typical warm forming flow curve showing a peak stress, a slight stress drop and a steady state region. As a general trend, a coarser initial grain size reduces the stress drop and shifts the peak stress and strain to higher values. Contrary, the stress–strain behavior at ambient temperature experiments is characterized by strain hardening and dynamic recovery.

The strain hardening versus stress curves in Fig. 3(b) illustrate the change in hardening behavior in more detail. At the beginning of deformation equivalent to small yield stresses, a higher hardening for the finer grained material was observed. Further straining changes the hardening behavior in the opposite direction, i.e. the finer grained material shows the stronger hardening for the 25 °C temperature. With increasing forming temperature this change is shifted to a smaller plastic strain or flow stress. Fig. 4 shows the influence of the strain rate on the stress–strain behavior at higher forming temperatures. Generally, for a lower strain rate a reduced flow-stress and a shift of the peak strain and stress to smaller values was observed. Differences in peak stresses and strains resulting from the initial grain size diminish with a slower strain rate and a higher forming temperature. A variation of deformation parameters to a lower Zener–Holloman parameter Z [temperature corrected strain rate ($Z = \dot{\epsilon}\, exp(Q/RT)$, where Q is the activation energy for warm forming and R the universal gas constant)] as well as a finer initial grain size changed the whole stress–strain kinetic from a single- to a multi peak flow behavior.

The hardening versus stress curves of Fig. 5 clearly show that the stage III of hardening and the point of inflection (stage III–V) is strongly influenced by the strain rate. At the point of inflection critical conditions (critical stress σ_c and strain ϵ_c) for a microstruc-