Mechanisms of New Grain Formation in a Ni-20%Cr Alloy during Warm to Hot Working

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Abstract. The process of new fine grain formation was studied in compression of a Ni-20%Cr alloy at temperatures ranging from 500°C (0.46T_m) to 950°C (0.73 T_m) at a strain rate of 7×10^{-4} s^{-1}. Two types of deformation behaviors with different features of microstructure evolution were observed, depending on processing conditions. The deformation behavior under flow stresses below about 500 MPa (relatively high temperatures) was typical for hot working associated with discontinuous dynamic recrystallization (DRX). The extensive local migration (bulging) of both initial and deformation induced high angle boundaries (HAGBs) resulted in the development of nuclei, which grew out leading to the formation of recrystallized structure with grain size of $D > 1$ μm. Numerous annealing twins were observed within these DRX grains. On the other hand, continuous DRX gave a major contribution to the formation of new grains at applied stresses above 500 MPa (relatively low temperatures). This fact was attributed to a low mobility of grain boundaries. The new grains with size of $D < 1$ μm were evolved due to gradual transformation of deformation induced low angle boundaries (LAGBs) into HAGBs.

Introduction

Warm to hot working accompanied by DRX is one of the most promising methods for processing of various metals and alloys [1-3]. The main regularities of DRX in metallic materials have been fairly clarified for hot working conditions, *i.e.* above ~0.7 T_m (T_m – melting point). The DRX is associated with the formation of new grains, the size of which depends on steady-state flow stress by a power law function. Under hot deformation conditions, the flow stresses can be related to the DRX grain sizes with a grain size exponent of about 0.7 [2].

The structural mechanisms of DRX are currently a topic for debates among materials scientists [2-6]. Discovered many years ago the bulging mechanism operates in materials with relatively low stacking fault energies during hot working [2, 3]. The local migration, bulging, of grain boundaries leads to the formation of nuclei, which then grow consuming work-hardened surrounding matrix. This process includes two sequential steps, *i.e.* nucleation and growth of DRX grains. The growing DRX grains experience a deformation upon further straining and then are taken up by new DRX nuclei. This process of sequential nucleation and growth leads to dynamically unchanged average grain size, the value of which depends on deformation conditions. Since the DRX microstructure evolving under processing actually consists of two structural components, work hardened grains and new recrystallized grains, such process is considered as a discontinuous phenomenon. On the other hand, the structural changes during hot working of metallic materials with high stacking fault energies are sometimes discussed in terms of continuous DRX [4]. The new grains appear as a result of a gradual increase in misorientations between deformation induced LAGBs upon straining. Therefore, the new grain development can be considered as one-step process.

The structural mechanism responsible for new grain evolution during deformation at relatively low temperatures have not been studied in sufficient details compared with that taking place during hot working. Recently, it has been suggested that the new ultrafine grains in various metallic materials develop without nucleation and growth after sufficiently large cold strains [7, 8]. In those cases, the mechanisms of microstructure evolution look like a type of continuous reaction.

The aim of the present work is to clarify the structural mechanisms leading to new grain evolution during hot to warm working. A Ni-20%Cr alloy was used as a typical single-phase metallic material having relatively high melting point and low stacking fault energy.

Experimental

A Ni-20%Cr alloy with a chemical composition of Ni–21%Cr–1.1%Si–0.3%Mn–0.75%Fe–0.31%Al–0.08%Ti–0.35%Cu–0.05%C and initial grain size of ~80 μm was used. The cylindrical samples of Ø10 × 12 mm were compressed by using a Schenck RMS-100 testing machine at temperatures ranging from 500 to 950°C and at an initial strain rate of 7×10^{-4} s^{-1} followed by quenching with water jet immediately after the termination of plastic deformation. The structural examinations were carried out on the specimens sectioned parallel with the compression axis by using a light microscope, a Quanta 200 and a Quanta 600 scanning electron microscopes fitted with automated electron back scattered diffraction pattern (EBSP) analyzers and an orientation imaging microscopy (OIM) system, and a JEM-2100 transmission electron microscope. The OIM images were subjected to a cleanup procedure, setting a minimal confidence index of 0.05. Detailed analysis of crystallographic orientations among fine (sub)structural elements was carried out by the TEM Kikuchi-line technique. The grain/subgrain sizes were measured on the TEM micrographs by the linear intercept method.

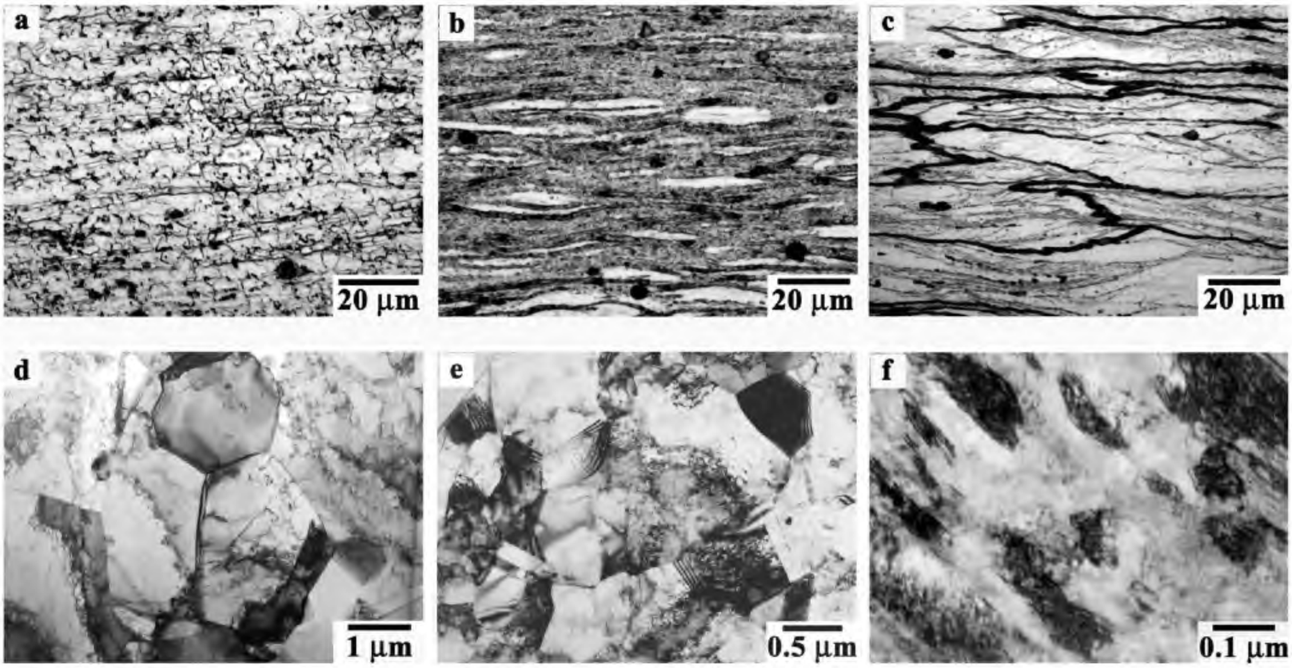

Fig. 1. Optical (a, b, c) and TEM micrographs (d, e, f) of deformed structures in a Ni-20%Cr compressed to a strain of 1.2 at various temperatures: (a, d) 900°C; (b, e) 700°C; (c, f) 500°C.

Results and Discussion

Deformed microstructures. The typical microstructures developed during deformation up to a strain of ~1.2 at temperatures ranging from 500°C to 900°C are shown in Fig. 1. It is clearly seen that the fraction of new fine grains depends significantly on the processing temperature. Therefore, these microstructures can be roughly considered as fully recrystallized (Fig. 1a), partially recrystallized (Fig. 1b), and cold-worked structures (Fig. 1c). The volume fraction of the new grains decreases from 70pct. at 900°C to 50pct. at 700°C and to a negligibly small value of 2pct. at 500°C. The new grains are characterized by a number of dislocations within their interiors (e.g. Fig. 1d and 1e). Thus, we can conclude that new grains resulted from DRX. The size of new grains strongly depends on the deformation temperature or the flow stress. A decrease in temperature from 900°C to 700°C leads to a decrease in the size of DRX grains from ~4 μm to <1 μm.

A plot on Fig. 2 demonstrates that the relationship between the DRX grain size (D) and the steady state flow stress (σ) can be approximated by power law functions:

$$\sigma = K\,D^{-N} \qquad (1)$$

where K is a constant and N is the grain size exponent. Two linear dependencies for the DRX grain size are clearly revealed in double logarithmic scale with inflection point around 500 MPa. The size of recrystallized grains can be related to the flow stress through a power law function with a grain size exponent of about 0.7 in the range of low flow stresses (relatively high temperatures), while the grain size exponent of about 0.25 was evaluated in the range of high stresses (relatively low temperatures). This bilinear relationship indicates the change in structural mechanisms responsible for development of DRX grains in a Ni-20%Cr alloy under different processing conditions.

Let us consider the DRX mechanisms operating at different temperatures. The samples processed at three specific temperatures, namely 900°C, 700°C and 500°C were selected for detailed examinations.

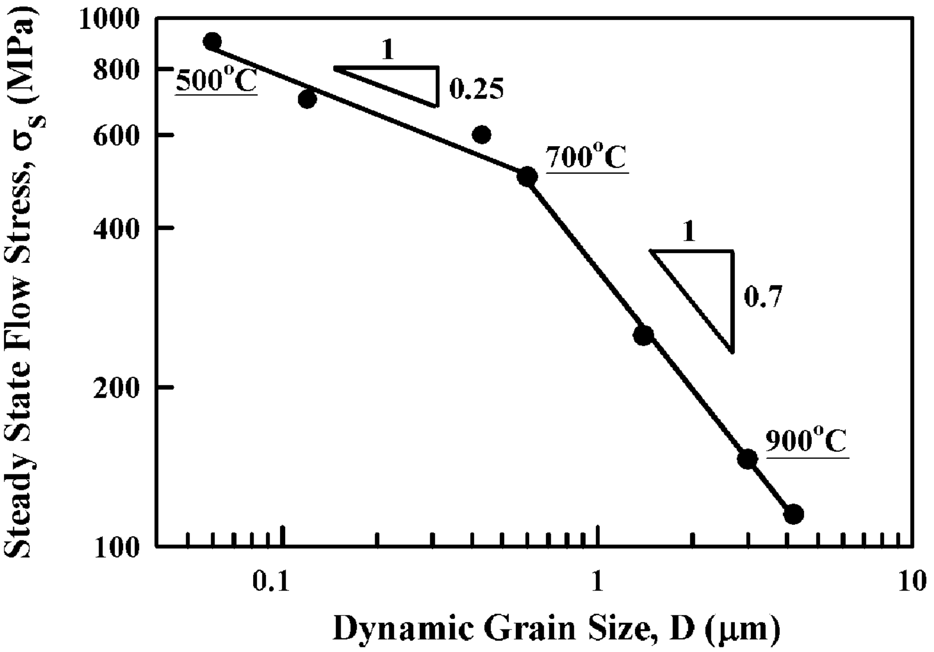

Fig. 2. Relationship between steady-state flow stress and size of dynamically recrystallized grains for a Ni-20%Cr alloy.

DRX at T = 900°C (0.7 T_m). In the range of hot deformation, the structural changes at an early stage of plastic flow are associated with the development of strain-induced subgrans. A number of LAGBs with misorientations less than 15° are evolved in vicinity of initial HAGBs (Fig. 3a). The dynamic nucleation occurs by a bulging mechanism. The local migration of initial HAGBs is accompanied by the formation of LAGBs, which separate the DRX nuclei from the parent grains. This leads to the development of fine DRX grains rapidly along the original boundaries and at triple junctions. The extensive bulging of both initial and newly developed HAGBs upon further straining results in the development of necklace-like microstructure that consists of chains of DRX grains surrounding the remainder of work-hardened matrix (Fig. 3b). Microstructure resulted from DRX includes the various grains, which correspond to different DRX stages, i.e. from the newly evolved nuclei with a size of about 1 μm being free of any LAGBs to work-hardened grains with relatively large sizes containing well-defined subgrains. It should also be noted that numerous annealing twins are observed within DRX grains. Such structural evolution is typical for discontinuous DRX [3, 5]. It can be concluded, therefore, that discontinuous DRX is the major mechanism of microstructural evolution operating in the Ni-20%Cr alloy during hot working under flow stresses below 500 MPa.

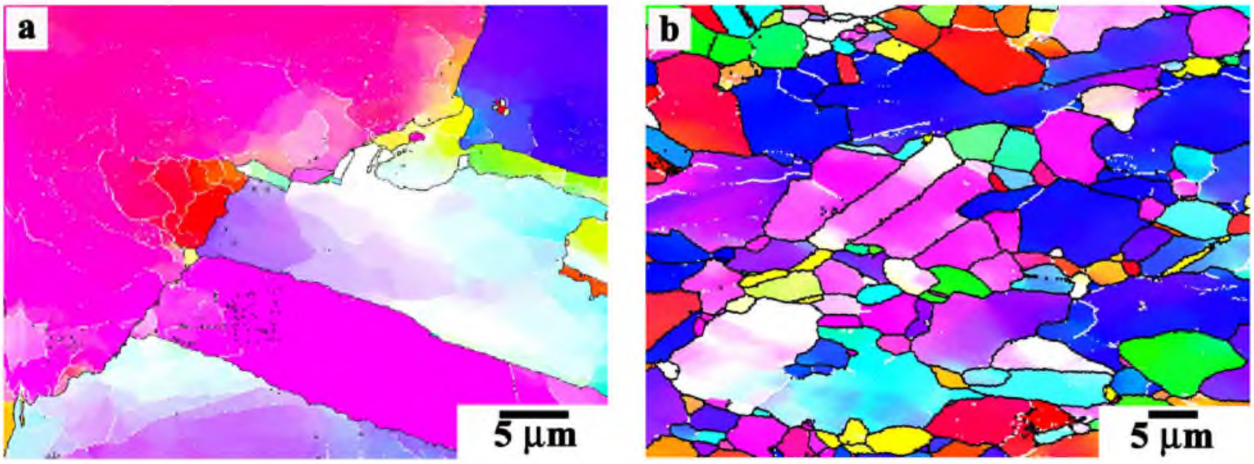

Fig. 3. Typical OIM micrographs for a Ni-20%Cr alloy deformed at 900°C to a strain of (a) 0.36, (b) 0.7. The boundaries with misorientations of $\Theta < 15^\circ$ and $\Theta > 15^\circ$ are indicated by white and black lines, respectively.

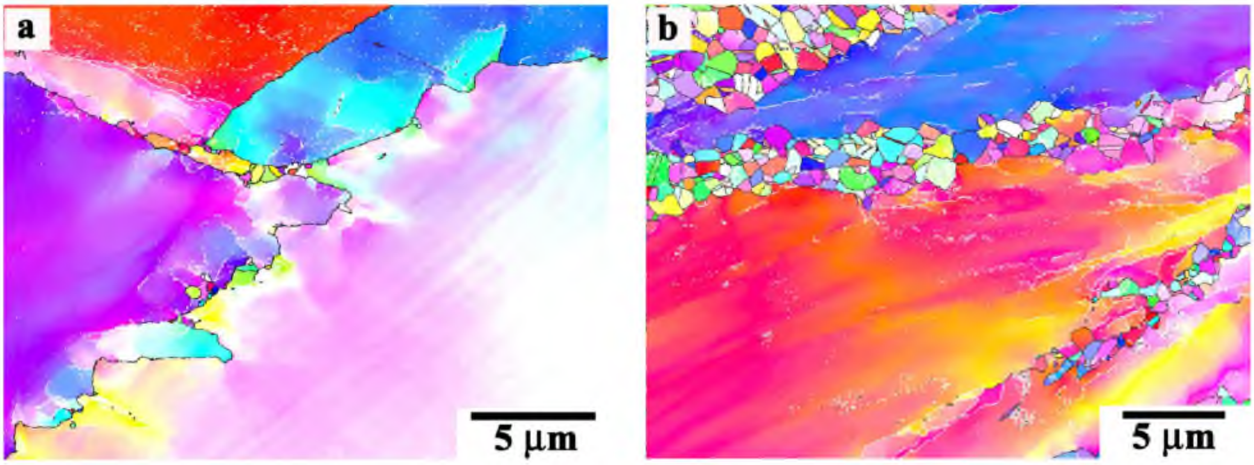

Fig. 4. Typical OIM micrographs for a Ni-20%Cr alloy deformed at 700°C to a strain of (a) 0.36; (b) 0.7. The white and black lines indicate the boundaries with misorientations of $\Theta < 15^\circ$ and $\Theta > 15^\circ$, respectively.

DRX at T = 700°C (~0.6 T_m). The recrystallized microstructure evolved at 700°C corresponds to the inflection point of two linear D - σ dependencies in Fig. 2. The features of structural changes associated with the new grain development at 700°C are quite similar with those taking place at higher temperatures. The first dynamically recrystallized grains develop at a strain of ~0.3 at initial HAGBs and triple junctions (Fig. 4a). It is clearly seen that new grains appear in areas of bulges along the highly serrated original HAGBs. Upon further straining, the fraction of DRX grains increases; and the new ultrafine grains with a size of 0.6 μm completely decorate the pre-existing HAGBs. In contrast to the samples processed at higher temperatures, kinetic of DRX process is slower. The necklace microstructure involving the recrystallized layers develops only at relatively large strains of ~0.7 (s. Fig. 4b) due to a small volume fraction of DRX grains. Therefore, the similar DRX mechanisms operate in the temperature range of 700 – 900°C. The decrease in the DRX grain size and the slowed DRX kinetics at lower temperatures are caused by slowing down diffusion rate and, hence, diffusion-controlled mobility of grain boundaries.

New grain formation at T = 500°C (~0.45 T_m). The microstructure after deformation at this relatively low temperature looks like a cold-worked one (Fig. 1c). However, detailed EBSP analysis reveals the formation of the new ultrafine grains (submicrocrystallites) near the original grain boundaries (Fig. 5a). It should be noted that plastic working to a strain of 0.7 results in incipient formation of recrystallized structure. Nevertheless, there exists a weak evidence for the formation of necklace structure at 500°C (Fig.5). In general, DRX hardly occurs under warm working at temperatures below 0.5 T_m. Suppression of discontinuous DRX at relatively low temperatures is related to extremely slow diffusion-related processes that inhibit the migration of both initial and deformation induced HAGBs over a large distance.

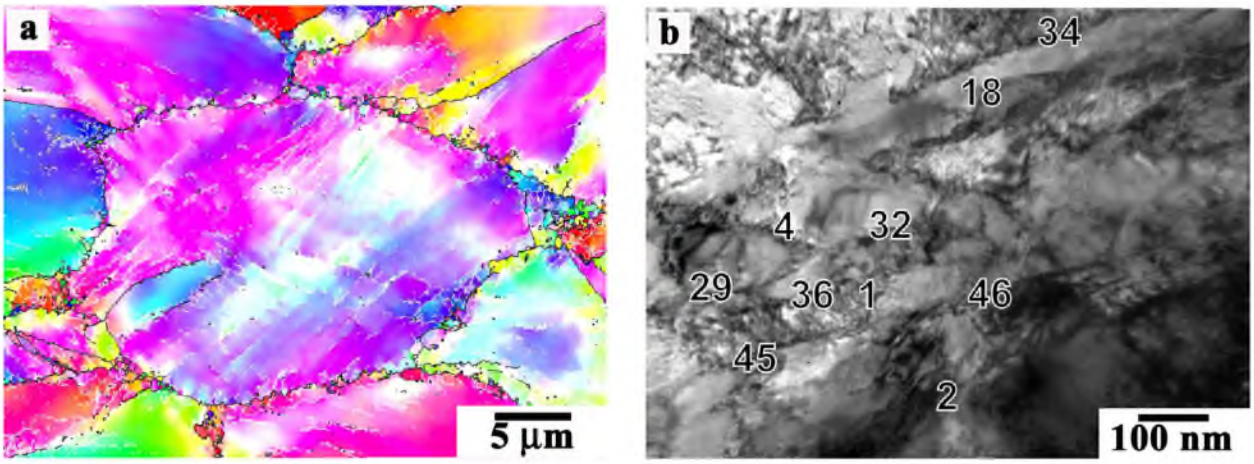

Fig. 5. Typical OIM image (a) and TEM micrograph (b) showing the new grains evolved near original grain boundaries in a Ni-20%Cr alloy strained up to 0.7 at 500°C. The boundaries in (a) with misorientations $\Theta < 15°$ and $\Theta > 15°$ are marked by white and black lines, respectively. The numbers in (b) indicate the misorientations in degrees.

Figure 5b shows a typical TEM micrograph of dislocation substructure that evolves near initial grain boundary at a strain of ~0.7. The microstructure in Fig. 5b looks like a severely deformed microstructure with extremely high dislocation density. Nanoscaled structural elements have an irregular shape and indistinct diffuse boundaries. Examination of the angular misorientations between these structural elements, however, revealed large misorientations typical for conventional HAGBs. The misorientations ranging up to 46 degrees were evaluated within the selected portion of microstructure in Fig. 5b. Therefore, these largely misoriented new structural elements that evolved during plastic working can be considered as new DRX grains. The size of such DRX grains (about 0.1 μm) formed at high stress is much finer than can be expected by extrapolation of data from low-stress region (see Fig. 2). The microstructural features described above suggest that the new grain evolution at temperature of 500°C is a result of progressive subgrain evolution. The accumulation of dislocation brings about the evolution of local angular gradients near grain boundaries [6]. This leads to the formation of large local misorientations and new grains. Such structural mechanism can be considered as a kind of continuous DRX [8].

Summary

The occurrence of DRX in a Ni-20%Cr alloy with initial coarse grained structure was examined during plastic working by compression at temperatures ranging from 950°C to 500°C (0.73 to 0.45 T_m) by means of EBSP and TEM analysis. The main results can be summarized as follows:

The relationship of steady state flow stress to the new grain size is expressed by a power law with the grain size exponent of ~0.7 in the range of low flow stresses (or high temperatures), while the grain size exponent of ~0.25 was evaluated in the range of high stresses (low temperatures). Such bilinear relationship is attributed to the change of DRX mechanism from discontinuous to continuous one with increasing flow stresses (decreasing temperature).

At low stresses (below about 500 MPa) the new grain evolution is associated with the grain boundary bulging: the local migration of both initial and deformation induced boundaries results in the development of recrystallization nuclei, which grow out leading to the new grained structure with grain size of $D > 1$ μm. Numerous annealing twins are evolved within growing dynamically recrystallized grains.

Continuous DRX gives a major contribution to the formation of submicrometer and nanoscale grains in the region of rather high stresses above 500 MPa. The new grains with size of $D < 1$ μm are formed due to evolution of deformation induced LAGBs, the misorientations of which increase with increasing strain.

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