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Equal-Channel Angular Pressing of NiAl

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Abstract. Equal-channel angular pressing (ECAP) was applied to polycrystalline NiAl at temperatures around the brittle-to-ductile transition temperature (BDTT). NiAl rods encapsulated in a steel jacket were ECAP-processed in a die with a channel angle of 120°. The microstructure and texture were characterized by electron backscatter diffraction with a scanning electron microscope. The volume fraction of the texture components typical for simple shear in the intersection plane of the channels changes in the range of the BDTT.

Introduction

NiAl, an intermetallic compound with B2 structure, shows a BDTT above about 300°C, which depends on different parameters, such as stoichiometry, alloying elements and grain size [1]. The main reason discussed for the BDTT of NiAl is the transition of secondary slip from {110}<111> and/or {112}<111> slip systems to {110}<110> [2]. Activation of these slip systems is necessary for polycrystalline plasticity, which requires five independent slip systems in order to fulfill the von Mises criterion [3]. Primary {110}<100> and/or {100}<100> slip only yields three independent slip systems. Above the BDTT, in the temperature range between 427°C and 827°C, slip activity along <100> and <110> during torsion, i.e. simple shear, leads to a main {110}<100> shear texture component [4]. To clarify, if the change of secondary slip from <110> to <111> at the BDTT also leads to a change in texture, ECAP has been applied. In ECAP [5, 6], one of the modern techniques to induce a severe plastic deformation (SPD), deformation mainly takes place as simple shear in the intersection plane of the two channels. Moreover, the application of backpressure during ECAP can assist in processing hard-to-deform materials, such as NiAl below the BDTT, as it increases the hydrostatic pressure in the shear zone and suppresses the formation of strain localizations and cracks. SPD below the BDTT will lead to a significant reduction of grain size. For NiAl it has been shown, that grain refinement may also lead to an increase in ductility [2].

Experimental and modelling details

Two cylindrical samples (diameter: 6 mm, length of sample 1: 60 mm, sample 2: 26 mm) cut by spark erosion from a hot extruded NiAl (49.8 at.%Al) rod were encapsulated into the center of 15 mm × 15 mm × 80 mm stainless steel jackets (AISI304). After closing the ends of the holes with copper plugs, the billet was preheated to 650°C and then within 10s transferred into the ECAP-die, heated to 185°C. The ECAP processing was performed with a punch speed of 3 mm/s in a die with an angle of 120° and sharp edges (Fig. 1). The friction conditions inside the die are optimized by two movable walls in the inlet channel and a bottom slider in the outlet channel. The moving parts and the billet were lubricated with MoS_2 grease. In order to suppress strain localizations and cracks, a backpressure of 300 MPa was applied. Assuming simple shear in the die, the shear strain γ after one pass is $\gamma = 2\cot(\phi/2) = 1.15$, with an intersection angle $\phi = 120°$ (Fig. 1). The processing temperatures were measured by several blind trials (without pressing) by a thermocouple in the centre of the billet according to the temperature decay of the pre-heated billet inside the colder die. This gave an average temperature of 300°C (325 to 275°C) and 270°C (280 to 260°C) for samples 1 and 2, respectively. It should be noted that the plastic work converted into heat is not considered. For more details of the ECAP processing see [7].

The microstructure and texture were determined by electron backscatter diffraction (EBSD, HKL) in a scanning electron microscope (SEM, Zeiss Ultra 55). The initial texture is a weak <110> fibre texture, which is typical for tensile deformation of B2 structures [8]. The ECAP microstructures and textures were taken from the centre of the flow plane of the samples (parallel to direction of processing). The step size used was 0.7μm and 0.4μm for the sample processed at 270°C and 300°C, respectively.

The EBSD patterns were post-processed by a personal orientation imaging software [11] and used as input in MTM-FHM software [9] to calculate the ODF. The Euler angles were used in Bunge notation [10]. To detect the grains with EBSD, once all the pixel-based boundaries were defined (as explained in the previous section), a flood-fill procedure was applied to search for sub-areas delimited by a closed boundary. Such sub-areas were considered as grains. The average grain size is calculated by an equivalent diameter circle area technique. The shear and ECAP sample coordinate systems are defined in Fig. 1. Because of monoclinic sample symmetry in one pass ECAP with axisymmetrical initial texture, the ODF representations are extended to $\varphi_1 = 180°$.

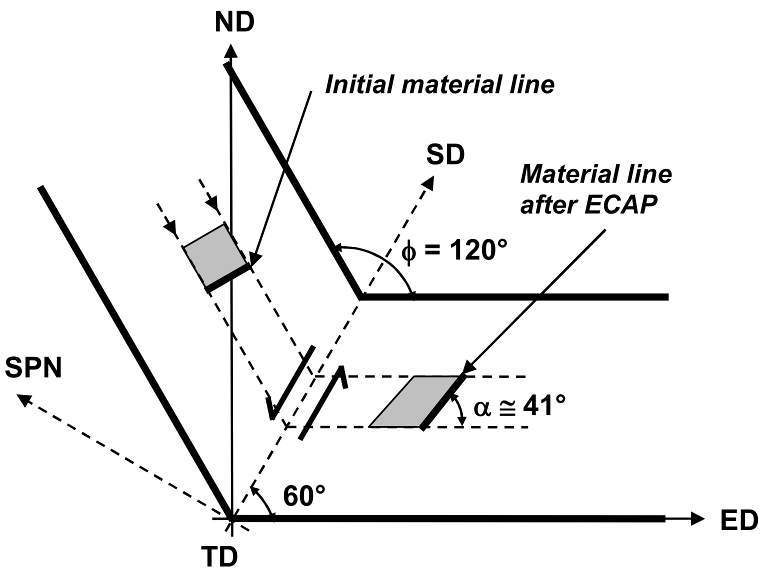

Fig. 1: Reference systems superimposed with volume element deformed by ECAP (finite element calculation). *Coordinate systems* used are: simple shear system with SD = shear direction, TD = transverse direction, SPN = shear plane normal, *ECAP system* with ED = extrusion direction, TD, ND = normal direction. After ECAP the structural elements have a theoretical inclination angle of 41° (see Eq. 1) with respect to ED.

Results and discussion

Figures 2a and b show EBSD maps with an inverse pole figure color coding of the two samples after extrusion at 270°C and 300°C, respectively. The misorientation between two measured points was calculated and a black line is plotted on the EBSD maps when it exceeds 5°, for smaller angles (between 3 and 5°) the boundaries are plotted in gray.

A material line entering perpendicular to the walls of the die appears rotated and elongated after ECAP. Assuming simple shear in the intersection plane, these lines display an angle α of about 41° with respect to ED (Fig. 1). This angle is expressed by:

$$\alpha = \frac{\pi}{2} - \arctan\left[2\tan\left(\frac{\pi}{2} - \frac{\phi}{2}\right)\right]. \tag{1}$$

It is marked by bold solid white lines in Fig. 2.

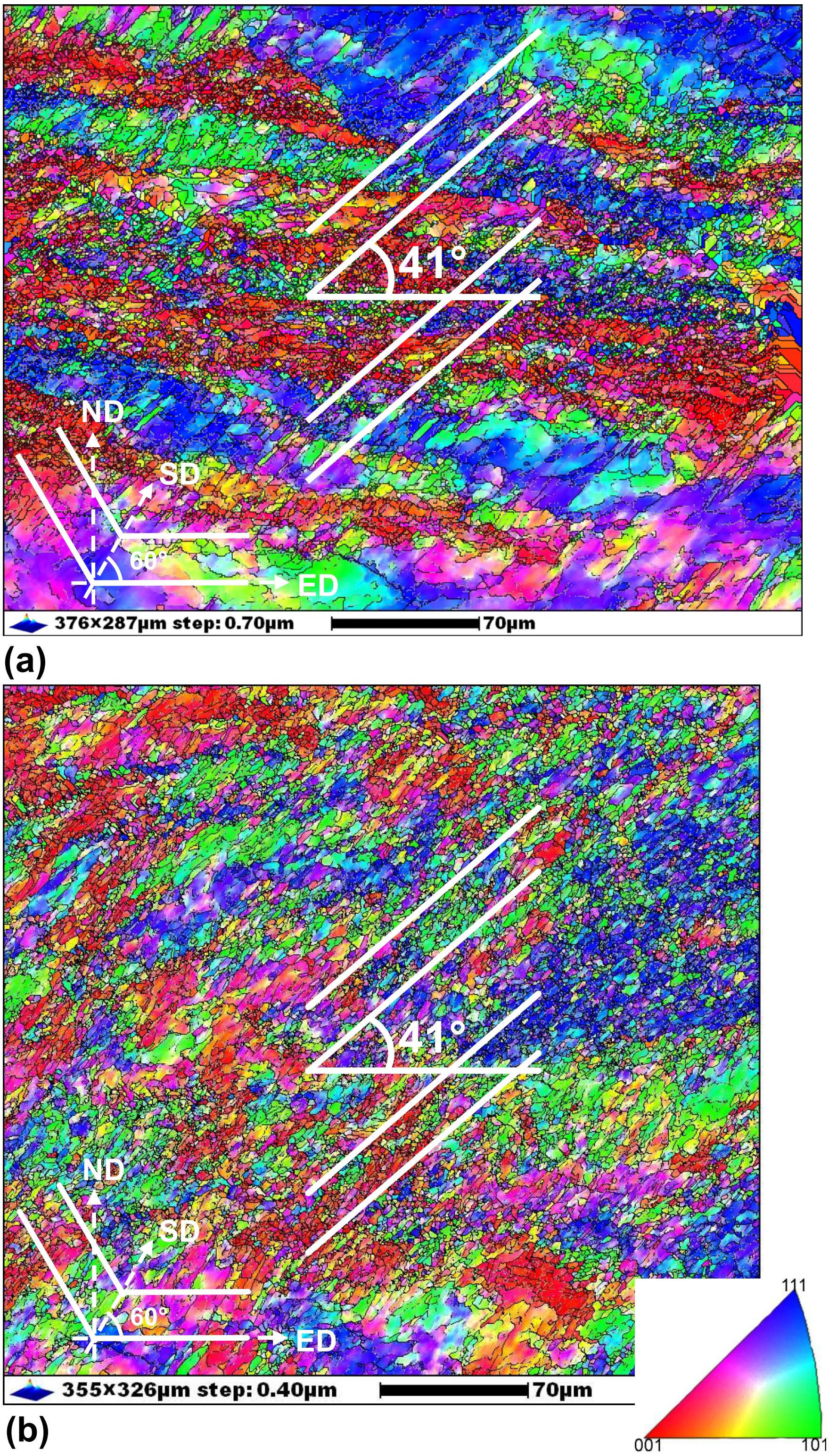

Fig. 2: Microstructures: (a) after ECAP at 270°C, (b) 300°C

The microstructures display fragmented regions composed of small grains as well as less fragmented ones with larger grains. They contain large internal misorientations and subgrain boundaries. To clarify if these grains are "fully" deformed by the ECAP process, the inclination and alignment of the subgrain boundaries can be used. As it can be seen, most of them are aligned according to the theoretical angle α. This suggests that those larger grains are deformed and that the amount of shear strain induced is close to the theoretical one. The small grains are assumed to have formed by continuous dynamic recrystallization during SPD [2, 4] in suitably oriented grains.

An important result is the effect of temperature on grain size. Figure 3 shows the grain size distributions for the initial sample and after ECAP at 270°C and 300°C. The overall average grain size for the initial material was 4.1µm, after ECAP the average grain size decreased to 1.4µm and 1.8µm at 270°C and 300°C, respectively. However, the distinction between the two samples is less evident from the grain size distribution graphs of Fig. 3. Most of the detected grains display a grain size of about 6µm for the initial material and 3µm for the deformed samples.

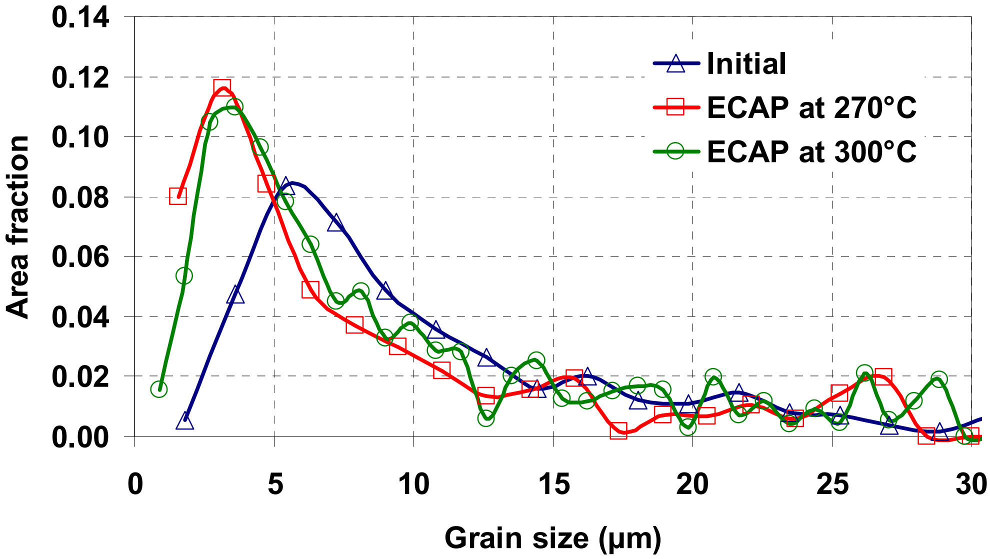

Fig. 3: Grain size distributions in the samples before and after ECAP

Figure 4 shows the texture of the two samples in the Euler space (Bunge notation [10], 1 = SD, 2 = SPN and 3 = TD) obtained from the EBSD measurements. In order to indicate the main rotation trends of the texture, the velocity field obtained from the simulation is also projected on the Euler space sections (black arrows), for more details on the velocity field see [12 - 14]. The texture is discussed in terms of ideal shear texture components which are displayed as black dots in the ODF sections. Both samples clearly display shear-type textures of bcc metals, but the ideal shear components of texture are formed with different intensities according the temperature of the ECAP-processing.

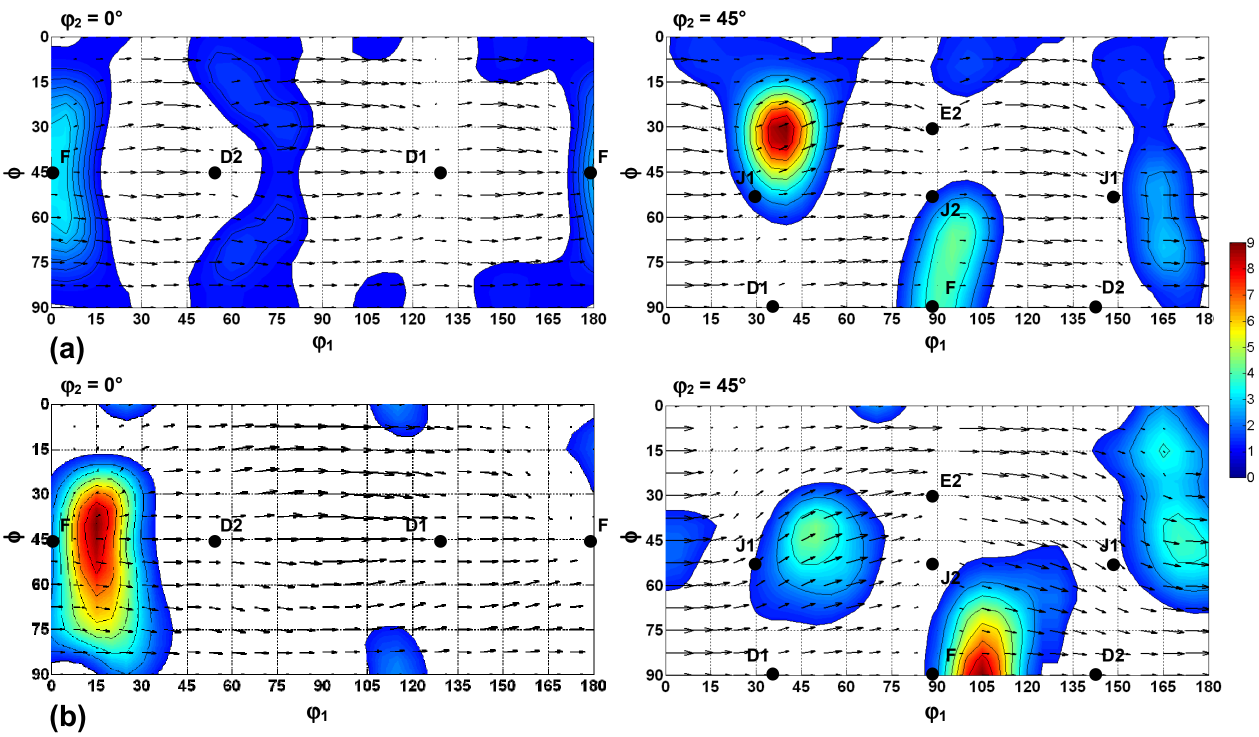

Fig. 4: Texture in the shear system represented in $\varphi_2 = 0°$ and 45° sections: (a) after ECAP at 270°C, (b) 300°C. The ideal shear components (black dots) as well as the rotation velocity field (black arrows) are given.

Figure 5 shows the change of volume fraction of the shear texture components with temperature. The EBSD measurement points disoriented by less than 20° with respect to all the components are considered to give a measure of the volume fraction of each ideal shear component. From Fig. 5 as well as from the texture (Fig. 4) it can be seen, that the F-component is dominant in the sample deformed at 300°C followed by the components of J1 and J2. A dominant F-component has also been observed in torsion above 427°C [4]. With decreasing temperature, at 270°C, J1 becomes the major component, twice as intense as F.

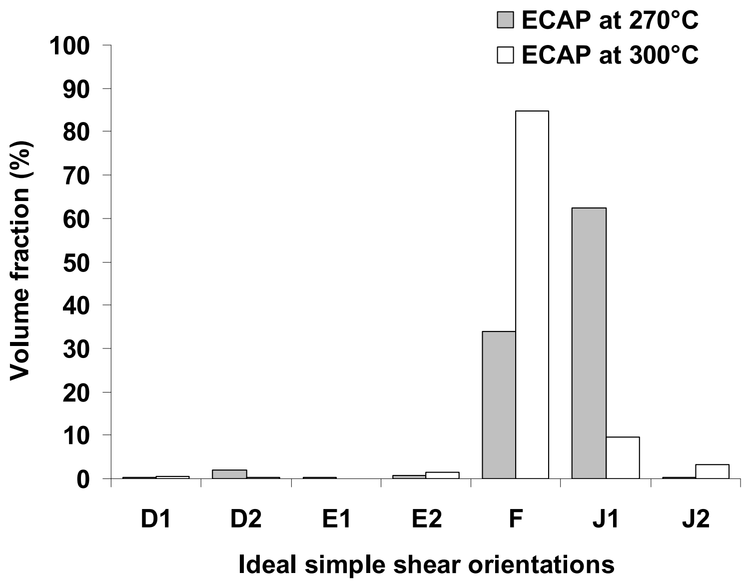

Fig. 5: Volume fraction of shear texture components for samples processed at different temperatures.

These preliminary results indicate a change of texture across the BDTT which seems to be related to the change in secondary slip [15]. Further experiments on the basis of ECAP-processing at lower temperatures as well as polycrystal texture modeling considering different slip system activity in the B2 structure are planned to support these assumptions.

Conclusions

1) NiAl can be processed by ECAP at temperatures in the region of the BDTT.
2) The initial microstructure becomes refined and develops typical shear features.
3) The texture changes from a <110> extrusion fibre texture to a shear texture.
4) The intensity of the shear components depends on the temperature of ECAP-processing.
5) The texture change seems to be related to the change in secondary slip at the BDTT.

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