Structural analysis of severely deformed Ni₃Ge by electron microscopy methods

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Keywords: High Pressure Torsion, Severe Plastic Deformation, L1₂ order, nanocrystals, chemical disordering

Severe plastic deformation of bulk materials leads to the formation of a nanocrystalline structure via grain refinement. It was shown recently that the formation of nanocrystalline structure induced by severe plastic deformation improves the room temperature tensile properties of brittle L1₂ structured Ni₃Al [1]. It is the aim of the present study to investigate the process of strain induced grain refinement in an L1₂ structured intermetallic alloy in detail starting from a single crystalline Ni₃Ge sample.

Single crystalline Ni₃Ge samples were severely deformed using high-pressure torsion (HPT). The HPT deformations of the discs (0.8 mm thick, 8 mm in diameter) were carried out at RT at a pressure of 6 GPa with 0.5, 5, 10, and 20 turns. Two sets of samples with different directions of the compression axis ([100] and [123]) were deformed. The deformation structure was investigated by transmission electron microscopy (TEM), scanning electron microscopy (SEM), electron backscattered diffraction (EBSD) and differential scanning calorimetry. EBSD measurements were carried out at different radii corresponding to different shear strain \( \gamma \). Due to the inhomogeneous deformation structure TEM samples were prepared from selected areas using focused ion beam method.

EBSD reveals that at low strains (0.5 turns, \( \gamma < 10 \)) the deformation leads to homogeneous fragmentation of the single crystal, and the misorientation between the fragments increases with increasing strain. SEM analysis shows that after 5 turns (\( \gamma = 90 \)) nanocrystalline bands (5 to 30 \( \mu \)m wide) occur and accumulate in regions within 100 \( \mu \)m near the top and bottom surface of the HPT disc. These bands evolve in both sets of samples. Figure 1 shows the presence of a thick nanocrystalline band on a cross section of the HPT disc (20 turns) using back scattered electrons. The EBSD measurement (shown as inset) reveals a highly fragmented band with high strain and a high density of defects since orientations within the band can hardly be identified. The area near the band shows a weak orientation gradient perpendicular to the band. Figure 2 shows a bright-field TEM image and a diffraction pattern of the interface region. The TEM analysis reveals three different structures occurring next to each other: (i) a highly deformed single crystalline matrix (SC) with a high density of defects accumulated on \{111\} planes, (ii) a textured nanocrystalline structure inside the bands (NC1) and (iii) a nanocrystalline structure with little texture (NC2) near the boundary of the bands in a region of about 3 \( \mu \)m thickness. The grains elongated parallel to the shear plane are about 20 nm wide and have lengths of around 100 nm. Combining the TEM results with differential scanning calorimetry, it can be concluded that the average grade of chemical disordering increases with increasing strain and is correlated mainly with the deformation induced increase of the density of bands.

In a nutshell, the analysis of strain induced grain refinement by electron microscopy methods yields a continuous homogeneous fragmentation of grains at low strains and the local evolution of nanocrystalline bands at larger strains. The orientation of the bands is correlated crystallographically whereas their position within the sample occurs in a random manner.
Figure 1. Ni$_3$Ge deformed by HPT (20 turns, $\gamma = 360$). Cross section SEM image, direction of compression [123]. Nanocrystalline bands (5 to 30 $\mu$m wide) occur in a random manner and accumulate near the surface of the HPT disc. The colored EBSD map indicates a weak orientation gradient perpendicular to the band.

Figure 2. Ni$_3$Ge deformed by HPT (20 turns, $\gamma = 360$); TEM diffraction pattern and bright-field image of a nanocrystalline band consisting of textured (NC1) and weakly textured (NC2) areas. The boundary of the nanocrystalline band lies parallel to a highly active $\{111\}$ plane of the single crystalline region (SC). In SC, the high activity is concluded by the accumulation of defects parallel to $\{111\}$ planes (dashed lines).

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