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Functional Materials Engineering Department	ID	079201
Name	CIUCA Octav Paul	

Advisor	Prof. Minoru UMEMOTO
	Prof. Hiroyuki TODA

Title	Process of disordering and grain refinement in Ni ₃ Al by high-pressure torsion
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(800 words)

Severe plastic deformation (SPD) techniques represent one of the most effective ways of producing ultrafine-grained structures in metallic materials, which showcase a wide range of improved mechanical and physical properties when compared to their polycrystalline counterparts. Among these methods, the high-pressure torsion (HPT) technique is applicable not only to pure metals and alloys, as with most other SPD techniques, but also to brittle materials such as a number of intermetallic compounds.

Ni₃Al is one of the most attractive intermetallics for HPT deformation, due to the improvement in ductility through disordering and in strength through grain refinement, both as a direct result of plastic deformation. Previous literature accounts suggest that HPT deformation of Ni₃Al leads to the formation of a duplex structure which includes coarse grains with partially-retained long-range order and a disordered nanocrystalline structure. The average grain size within the bulk is reduced with strain through a continuous change of the volume fractions of these two phases, until the entire structure becomes nanocrystalline. The formation of the nanocrystalline structure is highly localized, appears to be related to local disordering and occurs initially in the form of thin bands. However, the various reports of the mechanism of nanocrystallization are conflicting, specifically regarding the process of local disordering and of nanocrystalline formation, and the occurrence of deformation twins in the ordered structure. The present work attempts to elucidate these aspects, and also to analyze the impact of the microstructural changes brought on by the HPT process on the mechanical behavior of the material.

In the present study, the starting material was a cast and homogenized (1173K, 3.6 ks, furnace-cooled) binary Ni₃Al stoichiometric alloy, confirmed to be fully-ordered by X-ray diffractometry (XRD) analysis. Optical microscope (OM) observations revealed columnar grains of roughly 900 × 200 μm. Discs of 10 mm in diameter and 0.85 mm thick were prepared and deformed by torsion under an applied pressure of 5 GPa. The torsional deformation was carried out with a low speed of 0.2 rpm to minimize possible thermally-induced processes during the deformation process. The planar and diametric sections of the deformed samples were investigated by XRD, OM, SEM and Vickers microhardness testing. TEM samples were cut from

both the edge ($R \cong 3.5$ mm) and the center ($R \cong 0$ mm) of the as-deformed samples and electrothinned using a double-jet machine with an electrolyte of 20% H_2SO_4 in methanol at 253 K. Tensile specimens with a gauge of $1 \times 1 \times 0.5$ mm were spark-cut from selected samples, such that the center of the gauge was at $R \cong 1.5$ mm from the center of the HPT sample.

HPT processing leads within the first turn to an almost complete loss of long-range order of the deformed Ni_3Al structure. TEM investigations indicate regions of partially-ordered, fine fragments with very low misorientations. These have elongated block shapes and crystallographic boundaries along $\{111\}$ planes. Fine deformation twins form along these crystallographic boundaries; they are disordered, very thin (<20 nm), and cross the structure for lengths which occasionally reach several microns. Further deformation increases the density of disordered twins in the fragmented structure and also leads to secondary twinning along intersecting $\{111\}$ planes. This results in the fragmentation of the elongated blocks into more equiaxed fragments with crystallographic shapes and nanoscale sizes (<50 nm), and this structure is the precursor for the nanocrystalline structure.

The formation of the nanocrystalline disordered structure is observed in the earliest stages of deformation. It occurs as thin bands which separate the fragmented regions and form a very fine network throughout the volume of the sample. The nanocrystals are relatively equiaxed and have poorly-defined, non-equilibrium grain boundaries. Moreover, thin nanotwins are found in a large density within the nanocrystals, originating from the grain boundaries. These features of the nanostructure do not change significantly with further straining.

The volume fraction of the nanocrystalline phase increases preferentially in the midsection of the HPT sample parallel to the shear plane, leading to the formation of a thick band. The local densities increase gradually with the number of turns, causing an overall increase in the volume fraction of the nanocrystalline phase. This heterogeneous distribution at the macroscopic level is partly explained by the use of a constrained HPT setup in our experiments.

The early loss of long-range order with straining is accompanied by abrupt increases in hardness (~ 6.3 GPa, from ~ 2.3 GPa) and ultimate tensile strength (~ 2.23 GPa, from ~ 0.27 GPa), as well as by a substantial improvement in ductility. After 0.25 turns of HPT straining the structure exhibits plasticity under tensile uniaxial loading, in stark contrast to the brittle behavior of the starting structure. The initiation of torsion deformation results in a change of the fracture mode from brittle intergranular to ductile. The values of the mechanical properties become saturated in the early stages of HPT processing, despite the steady increase in nanocrystalline volume fraction with strain. This saturation can be explained by the formation of the nanocrystalline structure in the early stages of torsional straining, which may cause the change of the plastic deformation mechanism to a grain-boundary activated one.