

# Tensile properties and fracture behavior of nanocrystalline Ni<sub>3</sub>Al intermetallic foil

Z. Bojar,\* P. Jóźwik and J. Bystrzycki

*Institute of Materials Technology and Applied Mechanics, Military University of Technology, Kaliskiego 2, 00-908 Warsaw 49, Poland*

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Fully dense nanocrystalline Ni<sub>3</sub>Al intermetallic foil was successfully produced from as-cast coarse-grained sheet by heavy rolling at liquid nitrogen temperature and subsequent recrystallization. The mechanical properties and fracture behavior of the nanocrystalline foil with grain size about 20 nm were derived from uniaxial tension tests at room temperature. Typically, the nanocrystalline foil exhibits significantly higher yield strength (~2.6 GPa), and reduced tensile elongation (~4.0%) relative to its microcrystalline counterpart (yield strength ~0.7 GPa and tensile elongation ~22%).

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High strength Ni<sub>3</sub>Al intermetallic foil can be used for structural applications in the form of honeycomb structures, which have advantages in terms of being more lightweight, and having higher stiffness and higher strength than solid Ni-based superalloys. Recently, Hirano et al. [1,2] have successfully fabricated thin microcrystalline Ni<sub>3</sub>Al foils with thickness ranging from 57 to 315 μm by room-temperature rolling of sheets which were sectioned from directionally solidified ingots. They found that the tensile fracture strength of Ni<sub>3</sub>Al foils cold deformed to ~80% reduction is very high, 1.7–1.9 GPa, which is more than three times larger than that of as-directionally-solidified specimens. The tensile tested foils exhibit a low elongation to fracture after yielding below 1.0% and show local microplastic deformation by a dimple-like pattern on the fracture surface. As expected, recrystallized foils exhibit some ductility, 3–15%, in contrast to their cold deformed or as-cast counterparts. However, the yield stress after recrystallization dramatically decreases, even below 100 MPa [3].

More recently, we have shown that fully dense nanocrystalline Ni<sub>3</sub>Al intermetallic foil can be successfully fabricated by heavy rolling at liquid nitrogen temperature and subsequent annealing [4,5]. The nanocrystalline Ni<sub>3</sub>Al foil doped by zirconium and boron possesses ultrahigh yield stress (2.7 GPa) and ultimate tensile

strength (2.9 GPa) as well as enhanced elongation to fracture (3.1%). In this paper we present more details on the microstructure, room-temperature tensile property and fracture behavior of nanocrystalline Ni<sub>3</sub>Al foil fabricated by this method.

A nearly single-phase Ni<sub>3</sub>Al intermetallic alloy with the nominal composition of Ni–22.13Al–0.26Zr–0.1B (at.%) was induction melted from pure elements in high purity argon and cast into a steel mold. The obtained ingot, 60 mm in diameter and 150 mm in length, was cut along its axis into sheets about 8 mm in thickness using an electro-discharging-machine (EDM). Both surfaces of the sheets were mechanically polished. Subsequently, the sheets were cold rolled with liquid-nitrogen temperature (LN<sub>2</sub>T) cooling between consecutive rolling passes. The final degree of deformation defined as a percentage of cold work (%CW) was about 95%. The heavy deformed foil with a thickness of about 300 μm was subsequently annealed in the temperature range 600–700 °C for 15 or 30 min in an argon atmosphere. Following this processing route, it was possible to obtain large enough and sound rolled foils for subsequent tensile tests.

Figure 1 shows a typical Ni<sub>3</sub>Al foil with a thickness of 300 μm obtained by rolling at LN<sub>2</sub>T to 95% of CW. The foil was heavily work-hardened with a Vickers hardness of 660 HV0.1. The surface of the foil was crack-free and smooth with a shiny metallic luster.

The X-ray diffraction (XRD) measurements were carried out on a Seifert 3003 diffractometer using CuK<sub>α</sub> radiation at operating parameters of 30 mA, 50 kV

\* Corresponding author. Tel./fax: +48 22 683 9445; e-mail: [bojar@wme.wat.edu.pl](mailto:bojar@wme.wat.edu.pl)



**Figure 1.** Optical photograph of Ni<sub>3</sub>Al foil with a thickness of 300 μm obtained by rolling at LN<sub>2</sub>T to 95% of CW.

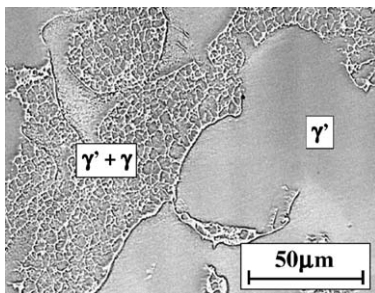
and a step size 0.02° per 3 s. From the registered XRD patterns the separation of grain size and lattice strain was carried out from Cauchy/Gaussian approximation by a linear regression plot [6].

The nanostructure of the specimens was investigated by using transmission electron microscopy (TEM). The TEM investigations were conducted on a Jeol JEM1230, operated at 120 kV. The thin foils for TEM investigation were prepared by jet electro-polishing.

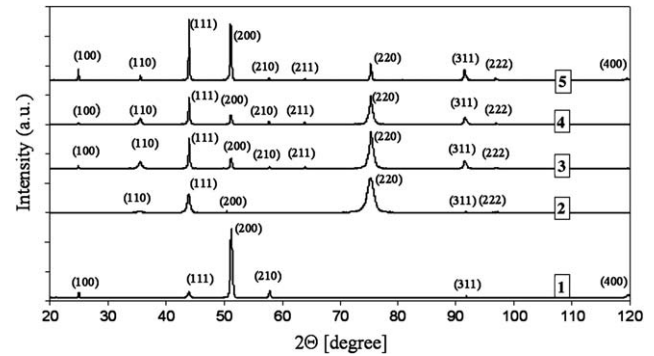
Tensile tests were performed in air at room temperature, at constant strain rate 10<sup>-3</sup> s<sup>-1</sup> using an Instron 8501 machine. Tensile specimens of nano- and micro-crystalline Ni<sub>3</sub>Al were cut from processed foils along rolling direction and then mechanically polished. The total length of the tensile specimens was 50 mm, the gauge length 20 mm, the gauge width 7 mm and the gauge thickness 0.4 mm. The fracture observations of tensile tested specimens were made on LEO 1530 field emission scanning electron microscope (FE SEM).

Figure 2 shows an optical micrograph of the as-cast Ni<sub>3</sub>Al intermetallic alloy, where large dendrites of ordered L1<sub>2</sub> Ni<sub>3</sub>Al (γ' phase) and discontinuous networks of disordered γ solid solution containing γ'-phase are visible. The γ phase was intentionally not removed by homogenization because the sheets sectioned from as-cast ingot could be more easily heavily rolled with LN<sub>2</sub>T cooling between consecutive rolling passes.

The X-ray diffraction patterns of Ni<sub>3</sub>Al-based alloy for selected processing routes are presented in Figure 3. It can be seen that the as-cast material is characterized by a high intensity of the (200) fundamental peak and the (100) superlattice peak, indicating the presence of a strong <100> texture (pattern no 1). Thus, the starting sheets for rolling were preferred <100> oriented columnar multicrystals. Borodians'ka et al. [2] have shown that the near <100> orientation in the directionally solidified γ'/γ ingot is desired for fabrication of crack-free foil by cold rolling.



**Figure 2.** Optical micrograph of as-cast Ni<sub>3</sub>Al intermetallic alloy.



**Figure 3.** X-ray diffraction patterns of Ni<sub>3</sub>Al intermetallic for selected processing routes: (1) as-cast; (2) LN<sub>2</sub>T rolling (95% CW); (3) LN<sub>2</sub>T rolling and annealing at 600 °C for 30 min; (4) LN<sub>2</sub>T rolling and annealing at 700 °C for 15 min; (5) room temperature (RT) rolling (60% CW) and annealing at 950 °C for 60 min (the reference specimen for XRD analysis).

After rolling at LN<sub>2</sub>T to 95% of CW, the broadened (220) Bragg peak appeared and the (200) and (210) fundamental peaks and the (100) superlattice peak almost vanished, indicating the formation of a strong (110) rolling texture (pattern no 2 in Fig. 3). The (110) superlattice peak is also observed but it is weaker than in the reference specimen (pattern no 5). The LRO parameter,  $S = 0.31$ , was estimated from comparison of the integrated intensities of the {110} superlattice peak with the {220} fundamental peak [7]. It indicates that the investigated material after LN<sub>2</sub>T rolling is in the partially ordered state. Similar behavior of the LN<sub>2</sub>T rolled Ni<sub>3</sub>Al doped by boron was observed by Ball and Gottstein [8]. It is in contrast to nanostructured Ni<sub>3</sub>Al processed by mechanical ball milling [9] or high-pressure torsion [10–14], where a complete loss of order was found.

Since the slip system in Ni<sub>3</sub>Al is <110>{111} at room temperature, it is likely that the {110} rolling texture is formed in the highly cold-rolled Ni<sub>3</sub>Al-based alloy. Hirano et al. [1,3] were found that room temperature rolled foil of stoichiometric Ni<sub>3</sub>Al single crystal has {110} texture, irrespective of the initial rolling direction and plane. There is also no large difference in cold-rolling texture between γ'/γ two-phase and γ' single-phase alloys [2]. Ball and Gottstein [8] carried out the most comprehensive study of microstructure and texture evolution in Ni<sub>3</sub>Al doped by boron during rolling at room and liquid nitrogen temperatures. They found that the rolling textures of Ni<sub>3</sub>Al + B deformed at liquid nitrogen temperature to 95% of CW are similar to the textures obtained at room temperature. The strongest component in a weak copper-type texture after large rolling reductions is the brass orientation ({110} <112>) and it is attributed to orientations inside shear bands. They did not observe dislocation cell formation and twinning even for deformation at liquid nitrogen temperature. The microstructure consisted of micro-band clusters as well as copper- and brass-type shear bands. The authors indicated that the rolling texture is strongly affected by the starting microstructure and can be quite inhomogeneous in coarse-grained material.

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