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Mechanical behavior of iron aluminides: A comparison of nanoindentation, compression and bending of micropillars

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ABSTRACT

Various local testing methods, namely, nanoindentation, compression and bending tests of micropillars were used to better understand the influence of ternary Cr atoms on the extrinsic and intrinsic mechanical properties of Fe_3Al intermetallics with the $D0_3$ super lattice.

Using such local techniques enables us to quantify the influence of Cr on the enhancement of the Young´s modulus. Furthermore, the effect of Cr on the yield stress, strain hardening and appearance of slip traces was studied based on the stress–strain curves and secondary electron micrographs of the bended and compressed pillars.

1. Introduction

Local mechanical testing methods such as nanoindentation, compression and bending tests of micropillars enable us to achieve an essential understanding of the deformation mechanisms of different materials [1–5]. The fact that they can be employed under a wide range of environmental conditions, i.e. at high (or low) temperature, in vacuum, harsh (or moderate) environments, allows the study of different deformation mechanisms close to routine industrial applications. In particular, these methods have been proven to be helpful in evaluating the influence of substitutional atoms [6,7] and/or interstitial atoms (e.g. hydrogen and nitrogen) [8] on the mechanical properties of different alloys. However, at the nano-micro scale, along with the intrinsic behavior of alloys (e.g. solid solution), extrinsic features (e.g. volume of deformed material or size effect (SE)) may influence strongly the dislocation assisted mechanical properties like yield and flow stress [9].

Nanoindentation has been used extensively for the last three decades to study the elastic behavior, incipient plasticity and post yielding behavior of different alloys [10–12]. In contrast to elastic properties, nano-hardness measurements have shown an indentation depth dependency in metals, i.e. the so-called indentation size effect (ISE). In the late 1990s, Nix and Gao [11] explained this ISE in terms of the storage of geometrically necessary dislocations (GNDs), caused by the local gradient in strain. The contribution of the density of GNDs (ρ_{GND}) along with the density of statistically stored dislocations (ρ_{SSD}) and the friction stress (τ_{P-N}) on the shear flow stress (τ_{flow}) was formulated for bcc metals as follows [10]:

$$\tau_{flow} = \tau_{P-N} + \varphi\mu b\sqrt{\rho_{SSD} + \rho_{GND}}, \tag{1}$$

where φ is an empirical factor depending on dislocation structures, b the Burgers vector and μ the shear modulus. The density of GNDs relates to the depth of indentation (h), geometry of the tip and magnitude of the Burgers vector as follows:

$$\rho_{GND}^{indentation} = \frac{3}{2bh}\tan^2\alpha. \tag{2}$$

Here, α is the angle between the surface of the indenter and the surface plane of the indented material [7,8]. Even though the nanoindendation technique presents several advantages considering its simplicity for studying a wide range of mechanical properties, it suffers from the complexity of the three-dimensional constraint form beneath the indenter tip [13].

The application of a uniaxial load (e. g. compression or tensile tests) avoids or reduces strain gradients to a great extent, thus

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facilitating the direct measure of the mechanical properties at the micrometer and sub-micrometer scale [9]. However, as in nanoindentation, a strong size dependency has also been observed in compressed or stretched micro samples [9]. Submicron- and micron-sized pillars have shown very high flow stresses, which scale with pillar size. This behavior has been related to the progressively harder operation of dislocation sources with decreasing size (source truncation) [14] and/or the reduction or even absence of dislocations in miniaturized samples and, hence, homogeneous or heterogeneous dislocation nucleation (dislocation starvation/ exhaustion) [14–19]. In particular, the size effect in bcc metals has been shown to scale with a ratio of test temperature to critical temperature and/or lattice resistance [20–22], which is strongly related to the low mobility of screw dislocations (SDs) leading to enhanced dislocation–dislocation interactions [23] or pile up of SDs in the vicinity of dislocation sources [21,24]. The threefold rotational symmetry of the SDs causes a non-planar core structure and associated Peierls potential that can be overcome by thermal activation [25,26]. In a uniaxial test, strong pile-up of dislocations may not be seen due to the homogeneous stress distribution in the micro-sample, especially in single-crystalline pillars, and hence, low work hardening may happen, which is far from the typical mechanical behavior of polycrystalline macro-scale samples.

More recently, *in situ* and *ex situ* bending of micropillars (beams) has been used to induce a non-uniform stress/strain field [14,27–29], with the characteristic that it enables the application of a tensile stress in addition to a compressive stress. Like in nanoindentation, the non-uniformity of the stress field leads to the storage of GNDs. Additionally, the existence of a very low number of available dislocation sources in an annealed material is responsible for their high activity, which leads to dislocation pile-up at the neutral plane. Dislocation pile-ups produce a back stress on the source, which may even shield the dislocation source and deactivate it after a certain period of time. In this case, a higher applied stress is necessary to re-activate the source, hence increasing the flow stress [30].

TEM measurements performed on Fe_3Al intermetallic with a bcc-based $D0_3$ superstructure show that the preferred slip system is $\{110\}\langle 111\rangle$. Due to the lack of closest-packed atomic planes, the screw dislocations cause the formation of a great number of dipoles in the deformed areas [31,32]. Short distance double cross-slip onto $\{112\}$ planes and back onto $\{110\}$ planes has been observed earlier [31,32]. This produces wide slip bands in localized areas at temperatures below 350 K, while adjacent areas remain free of mobile dislocations. However, the existence of anti-phase boundaries (APBs) that connect the $b = a/4\langle 111\rangle$ super partials may enhance the slip planarity in comparison to bcc metals [33].

In the present work, the nanoindentation, compression and bending techniques are employed to study the deformation mechanisms and the effect of Cr as a ternary alloying element on the elastic and plastic properties of $D0_3$ iron aluminides. More importantly, the abilities of each technique for characterization of various mechanical properties are critically compared.

2. Experiments

2.1. Sample preparation

Fe-26Al and Fe-26Al-5Cr intermetallics were produced by the Max Planck Institute for Iron Research in Düsseldorf using the induction melting method in an argon atmosphere. In both samples, the concentration of aluminum remained constant at 26 at%. In the ternary alloy, 5 at% Cr was substituted for iron atoms. Different cylindrical samples with a nominal diameter of 12 mm were cut using the spark erosion method. The samples were then heat-treated at 1200 °C for 24 h in vacuum with subsequent furnace cooling for homogenization. Afterwards, the homogenized samples were annealed at 400 °C for 168 h in a $\approx 10^{-6}$ atm vacuum to achieve a $D0_3$ structure with grain sizes larger than 500 μm in diameter.

2.2. Metallographic preparation

For all samples, the working surface was ground with up to 4000 grit emery paper, and subsequently cleaned, first by using ethanol and then by submerging them for ten minutes in a 50% ethanol–50% isopropanol ultrasonic bath. The samples were polished afterwards with successively finer diamond suspensions from 3 to 0.25 μm. Finally, since a low surface roughness and low dislocation density was a prerequisite for our measurements, the samples were electro-polished in a $1MH_2SO_4$ – methanol solution in order to remove the mechanically damaged layer.

2.3. Characterization of samples

Quantitative and qualitative characterizations of the samples were conducted using the following methods:

2.3.1. X-ray diffraction (XRD)

X-ray diffraction was performed in air, using a PANalytical X'Pert Pro MPD diffractometer. The X-rays were generated using a 40 keV accelerating voltage and a 40 mA current, with a copper target. XRD measurements on several binary and ternary alloys showed the existence of $D0_3$-ordered, single phase solid solutions at room temperature (Fig. 1a).

2.3.2. Scanning electron microscopy (SEM)

A Zeiss ΣIGMA™ -VP Field Emission Scanning Electron Microscope (FE-SEM), equipped with energy dispersive X-ray (EDX) and electron backscatter (EBSD) detectors, was used to characterize the different samples. Additionally, Oxford Instruments AZtec® EBSD system combined with Nordlys hardware was used for performing EBSD measurements. After EBSD measurements (Fig. 1b), a grain with the orientation close to the (001) in surface normal direction was selected. Afterwards, all tests, consisting of nanoindentation, compression and bending of micropillars, were performed in the defined crystal orientation.

2.4. Focused ion beam (FIB) milling

Micropillars were machined with an FEI Versa 3D Dual Beam microscope with a liquid gallium ion source. The annual milling procedure [34] was employed to cut pillars with a radius of approximately 0.5 and aspect ratio of 6:1 (height: diameter). This method consisted of a first coarse milling step done using a circular ring pattern with an outer diameter of 30 μm and an inner diameter of 2 μm. This was carried out at a beam voltage of 30 kV and beam currents of 15 nA. Second, multiple fine milling steps were performed at low currents between 30 and 100 pA to decrease the ion damage and fine-tune the size of the pillars. The aspect ratio of the compressed and bended pillars was the same, and the pillars were slightly tapered along the column. The top radius of all pillars was between 0.50 and 0.52 μm, the bottom radius was between 0.82 and 0.85 μm and the length of the pillars was between 6.00 and 8.40 μm. The measured tapering angle was less than 3° for all pillars.

2.5. Nano-micro indentation

Nano-indentations (NI) were performed with a Hysitron TI 900 Tribo-Indenter equipped with a Performech controller and a nano-