Mechanical characteristics under monotonic and cyclic simple shear of spark plasma sintered ultrafine-grained nickel

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ABSTRACT

The present work focuses on understanding the mechanical behavior of bulk ultrafine-grained nickel specimens processed by spark plasma sintering of high purity nickel nanopowder and subsequently deformed under large amplitude monotonic simple shear tests and strain-controlled cyclic simple shear tests at room temperature. During cyclic tests, the samples were deformed up to an accumulated von Mises strain of about $\varepsilon_{VM} = 0.75$ (the flow stress was in the 650–700 MPa range), which is extremely high in comparison with the low tensile/compression ductility of this class of materials at quasi-static conditions. The underlying physical mechanisms were investigated by electron microscopy and X-ray diffraction profile analysis. Lattice dislocation-based plasticity leading to cell formation and dislocation interactions with twin boundaries contributed to the work-hardening of these materials. The large amount of plastic strain that has been reached during the shear tests highlights intrinsic mechanical characteristics of the ultrafine-grained nickel studied here.

1. Introduction

Ultrafine-grained (*ufg*) metals and alloys are materials having grain sizes (*d*) in the 100 nm–1 μm range and exhibit a high room temperature (RT) yield strength [1,2] compared to conventional materials with large grains in accordance with the Hall–Petch (HP) relationship [3,4]. Unfortunately as shown by the body of the existing literature data [5–8] the observed high yield strength is usually accompanied by a lack of tensile ductility during monotonic testing of these materials, especially within the lower grain size range of *ufg* materials. This is particularly true for powder metallurgy (PM) processed materials because of processing defects such as impurities or incomplete particle bonding. Despite the processing of specific microstructures such as nanotwinned Co [9] or Cu [10] processed by electrodeposition that exhibit an appreciable tensile ductility at RT, studies reporting on the deformation mechanisms of *ufg* polycrystals at finite strains are scarce. This paucity therefore limits the use of this promising class of materials despite strategies that have been suggested to increase their ductility [11].

Cyclic loadings are of particular interest for many structural applications. Limited studies have been conducted on *ufg* Al [12,13], Ti [14] and Cu [15,16], mostly processed by equal channel angular pressing because of its capability to produce contaminants and porosity-free *ufg* materials in bulk form. In majority, these studies

concerned with small amplitude strain- or stress-controlled tests in both low-cycle and high-cycle fatigue regime and valuable data have been gathered. For instance, in the case of *ufg* Al, it has been shown that the number of cycles to failure of the as-processed and annealed samples was far below that of the coarse-grained (*cg*) counterpart [12]. In addition the stress–strain curve showed energy dissipation that occurred in the course of straining, which is never observed for the *cg* material counterpart [12]. For ECAP-processed Ti, no cyclic softening or degradation during the strain-controlled experiments was noticed opposed to what was reported for wavy slip materials such as Cu [16]. Furthermore, rapid cyclic instability, severe damage localization and premature fatigue failure were reported whose origin was related to the microstructure instability of the ECAP-processed *ufg* metals [15]. In the same context, the Bauschinger effect during uniaxial loading and unloading of *ufg* Cu has been investigated [14,17]. The influence of the heat treatment on the saturation stress, the hardening evolution and tensile–compression asymmetry, have been reported.

Nickel is one of the most studied metallic materials and an abundant experimental and theoretical literature exists on the deformation mechanisms of nanocrystalline (nc) and *ufg* Ni processed by different routes and deformed via various mechanical tests in the quasi-static strain rate regime at RT [18–25]. In addition, *ufg* nickel is a promising material for applications in such a field as micro-electro mechanical systems (MEMS) [24,26,27], for which cyclic properties can be considered as key issues. Therefore, for reliability matter, it is important to analyze mechanical properties *versus* microstructure relationships of *ufg* microstructures

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by examining the material behavior at finite strain. Indeed, to the best of the authors' knowledge, no study reporting on the mechanical behavior and the underlying deformation mechanisms under large amplitude deformation-controlled monotonic simple shear, nor on large amplitude deformation-controlled cyclic simple shear has been conducted on this material, especially in the *ufg* regime. Contrariwise to microstructures processed by severe plastic deformation (SPD) and in spite of some inherent drawbacks, powder metallurgy routes yield stable and texture free bulk *ufg* specimens [1,28]. Nonetheless, as it is the case of the majority of *ufg* materials, it has been shown that the RT ductility of the microstructure from this type of process is low [1,2,29].

In the present work we report on the mechanical behavior and the underlying deformation mechanisms during large amplitude strain-controlled monotonous and cyclic simple shear tests at RT of *ufg* nickel specimens processed by spark plasma sintering (SPS).

2. Experimental procedures

High purity (99.99%) Ni nanopowder with a nominal particle size of 100 nm was supplied by Argonide Corporation (USA). The powder was produced by electro-explosion of Ni wires [30,31]. The bulk samples were processed by spark plasma sintering (SPS) route using a Sumitomo 3rd generation apparatus, located at the CNRS platform facility at CIRIMAT (Toulouse). The principles of SPS have been reported elsewhere [32]. Before SPS-processing, the capsule containing the powder was broken in air and rapidly transferred to the graphite mould. The powder was held under a pressure of 150 MPa at 500 °C for 4 min (sample A) and 2 min (sample B) while pulses of high current density were applied to the sample for promoting consolidation. After SPS, disks of 50 mm in diameter and ~3.15 mm (sample A) and ~2.85 mm (sample B) in thickness were obtained.

After processing, the densities of the as-processed bulk samples were determined by the Archimedes method [1]. The computed relative densities of the samples were 95.5 ± 0.1% and 97 ± 0.1% for samples A and B, respectively. Given that both samples have been processed under the same conditions (except for the duration of the SPS experiment), the observed difference of the relative densities between the two samples is partially due to the efficiency of the sintering process relative to the amount of powder as the amount of powder for the processing of sample B was lower (therefore less initial porosity) than that of sample A. In addition, the slightly higher amount of face centered cubic NiO phase (having lower density, 6.67 g/cm^3, than that of Ni) in sample A revealed by X-ray diffraction investigation as described below may also contribute to the lower relative density of sample A.

The phase contents were further studied by X-ray diffraction (XRD) using a Philips Xpert powder diffractometer with CuKα radiation [28]. Further, the microstructure of the consolidated and the deformed samples was studied by X-ray line profile analysis using CoKα$_1$ radiation. The scattered X-rays were detected by imaging plates with the angular resolution of 0.005° in Θ, where Θ is the angle of diffraction. The line profiles of Ni phase were evaluated using the extended Convolutional Multiple Whole Profile (eCMWP) fitting procedure as described in detail elsewhere [33,34]. This method gives the crystallite size, dislocation density (ρ) and the twin-fault probability (β) with good statistics. The twin-fault probability is defined as the fraction of the faulted {1 1 1} planes along their normal vector. To complement XRD investigations, the microstructure of the bulk samples was also investigated by electron backscatter diffraction (EBSD) on a Leo S340 scanning electron microscope (SEM) and by transmission electron microscopy (TEM) using a JEOL-2011 electron microscope at an operating voltage of 200 kV. Before EBSD analysis, samples were electropolished with

A2 electropolishing solution from Struers at a voltage of 12 V for 15 s. EBSD analyses were carried out using a step size of 0.2 μm. Specimens for TEM investigations were first thinned mechanically and dimpled to about 20 μm and followed by final thinning using a Gatan Precision Ion Polishing System (PIPS) until perforation. More details regarding the experimental procedure have been given elsewhere [28].

The mechanical behavior of the sintered samples was studied by monotonic, cyclic load/unload, Bauschinger simple and cyclic Bauschinger simple shear tests, conducted at RT. These tests were carried out by means of a shear device mounted on a MTS M20 testing machine bearing a maximum load capacity of 100 kN. The simple shear specimens were rectangular plates of 30 mm length and 18 mm width cut through the thickness of the disks by electro discharge machining (EDM) to avoid surface hardening. Fig. 1a and b shows the location of the specimens in the compacted disks used in different mechanical tests. Specimens A1–A3 were subjected to monotonic simple shear test, while samples A4, A5 and A6 were used in cyclic Bauschinger simple shear test, simple shear load/unload test and Bauschinger simple shear test, respectively. Specimens B1, B2, B3 and B4 were subjected to Bauschinger simple shear, monotonic simple shear, cyclic Bauschinger simple shear and simple shear load/unload tests, respectively. Standard mechanical polishing using SiC papers and diamond paste was conducted in order to (i) remove the surface layer which might be remelted due to EDM and (ii) achieve a smooth surface free from stripes to reduce crack initiation during mechanical tests. The gauge area is limited to 30 mm × 2 mm. The final sample thickness was about 1 mm. It is worth noticing that the samples taken from the mid-thickness of the compacted disks displayed some porosity that was revealed after polishing, particularly for sample A. The planar simple shear test device consists of two rigid bodies, one of which is subjected to a translational movement relative to the other. The specimens were firmly fixed on the two bodies by two pairs of grips. A sketch of the shear device and specimen clamping is shown in Fig. 1c. All simple shear tests were performed using a constant plastic shear rate 1.732×10^{-3} s^{-1} (which corresponds to an equivalent von Mises strain rate of 10^{-3} s^{-1}). More details about the simple shear tests have been given elsewhere [35,36].

3. Results

3.1. Microstructure characteristics of the as-processed samples

The characteristics of the microstructure of the as-processed samples are summarized in Table 1. The dislocation density and the twin probability are two times higher for sample A in comparison with sample B. For a given technique used, TEM or XRD, it should be noticed that the grain/crystallite size values are the same within the experimental error for the two as-processed samples. In addition, the average grain sizes of both samples determined by TEM (by counting more than 200 grains) were about ~306 nm for sample A and ~318 nm for sample B. This is, in accordance with previous reports [37], three times larger than the size of the coherently diffracting domains measured by XRD profile analysis (~120 nm). The intensity ratio of NiO and Ni peaks at $2\Theta = 37.4°$ and $44.6°$ indicates that the relative oxide content is slightly higher for sample A (0.6%) than for sample B (0.5%).

Fig. 2a shows a typical EBSD scan (presented is a part of a 100 μm × 100 μm grid) of the as-processed microstructure in sample A and reveals that while no preferential grain orientation was found, the microstructure is heterogeneous and consists of a matrix of *ufg* grains within which micrometer-size (*mc*) grains are embedded. Some of these *mc* grains are subdivided into smaller grain blocks while still keeping more or less their initial spherical shape.