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Investigation of deformation micro-mechanisms in nickel consolidated from a bimodal powder by spark plasma sintering

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ABSTRACT

Bulk polycrystalline nickel compact was processed by spark plasma sintering from heterogeneous powder consisting of a mixture of nanometer and micrometer sized particles. The consolidated samples inherited the bimodal structure of the starting powder and was composed of ~55 vol.% coarse-grained (with the grain size larger than 1 μm) and ~45 vol.% ultrafine-grained (with an average grain size of ~550 nm) components. The deformation mechanisms were established by EBSD, X-ray line profile analysis and in-situ TEM observations. In the ultrafine-grained volume, the deformation occurred mainly through the activation of dislocation sources emitting full or partial dislocation either from grain interior or grain boundaries. Besides dislocation activity, rolling and sliding of nanograins were also observed during deformation by in-situ transmission electron microscopy, which have a considerable contribution to the observed high strain rate sensitivity of the bimodal microstructure. The cracks formed during deformation easily propagated in the nanograin regions due to the weaker particle bonding caused by the relatively high fraction of native oxide layer on the surface of the initial nanoparticles.

1. Introduction

Microstructures with enhanced mechanical properties are generally obtained by means of grain size reduction, development of multi-modal grain size distribution, and tailoring grain boundary (GB) structure. Indeed, it is well known that nanocrystalline (NC) and ultrafine-grained (UFG) materials have superior mechanical strengths compared to their conventional coarse-grained (CG) counterparts [1–4]. Among the several ways of processing of bulk materials with NC or UFG microstructure, the compaction of powders [1–5] has a fundamental place due to its versatility in tailoring in-demand composite-like microstructures. This type of process enables to obtain centimeter-scale and fully dense materials with different grain sizes spanning the nanometer (30–100 nm) range to the UFG (100–1000 nm) regime and above [3,4].

Recently, much attention has been paid to the novel spark plasma sintering (SPS) consolidation technique [6,7]. The most important advantages of the SPS processing route compared to conventional consolidation methods are the much shorter duration and the lower temperature of processing, which limit grain-growth during consolidation [8].

NC materials have so far demonstrated enhanced mechanical characteristics, such as high strength and fracture stress. Nevertheless, other key properties such as ductility, which is very important in forming processes, still needs to be improved. In that context, different strategies have been proposed to offset the low tensile ductility accompanying the high flow stress of NC and UFG materials [9,10]. For instance, it has been demonstrated that controlled volume fraction of twins in Cu induces both high strength and considerable tensile ductility, the latter being due to the strain rate sensitivity improvement by the presence of coherent twin boundaries (TBs) [11–14]. Another strategy for the improvement of ductility of NC and UFG materials is the incorporation of coarse grains in a fine-grained matrix [9,15–17]. In this case, the improvement of ductility can be attributed to the larger dislocation activity (mobility and storage) in coarse grains, thereby enhancing the strain hardening capability of materials [18]. The bimodal grain-structure can be achieved by heterogeneous grain-growth in severely deformed materials [19–21] or via abnormal grain-growth caused by microstructure heterogeneities such as inhomogeneous solute distribution and/or residual porosity [22]. Although in some cases the magnitude of the improvement of ductility does not meet the high expectations raised [23], the processing of bimodal microstructures is still often suggested in order to improve the mechanical response of metallic materials [9,18,24,25]. Our previous studies have demonstrated the successful processing of bimodal microstructures in Ni by SPS [17] and studied their mechanical performance under impact loading [25]. However, the deformation mechanisms in the CG and UFG volumes during quasistatic deformation have not been identified yet.

In the present work we investigate the room temperature (RT) quasistatic plastic behavior of an SPS-processed polycrystalline Ni

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with bimodal grain-structure at different strain rates. The underlying deformation mechanisms are revealed by post-mortem EBSD and X-ray diffraction (XRD) experiments, as well as by in-situ TEM investigations. The results may yield a better understanding of the contributions of the different components in a heterogeneous grain structure to the mechanical performance.

2. Materials and procedures

2.1. SPS processing

Bulk Ni samples were consolidated by SPS. The starting high purity (>99.8 wt.%) Ni powder was fabricated by electro-explosion of wire (Argonide Corporation, Stanford, USA) [26]. The sintering of the powder during SPS is assisted by the simultaneous application of direct current pulses of very high intensity (several thousands of amperes) and a uniaxial pressure exerted on the encapsulating system [27]. In the present case, the Ni powder was sintered by the SPS apparatus (model 515S-SYNTEX) located at the regional SPS platform facility hosted by ICMPE (Thiais, France). More details have been given elsewhere [28]. For the present study, SPS processing was carried out at a temperature of 600 °C for 15 min. The dwell pressure during sintering was set as 100 MPa. The relative density of the bulk sample was evaluated by the method based on the Archimedes principle and yielded a value of about 98%.

2.2. Mechanical properties

Following sintering, the macroscopic mechanical behavior was studied by means of uniaxial compression at room temperature and two different strain rates of $2 \times 10^{-4}\,s^{-1}$ and $2 \times 10^{-2}\,s^{-1}$ in order to investigate the effect of strain rate on deformation behavior. For compression tests, prismatic samples (3 mm × 3 mm × 5 mm) were mechanically cut from the compacted material and tested using an Instron universal machine (model 1195) with a 10 kN maximum capacity load cell. The strain was computed from the crosshead displacement corrected for the stiffness of the machine.

2.3. Microstructure study of the as-consolidated specimen and post-mortem analyses of the deformed sample

The microstructure of the as-consolidated and the subsequently deformed Ni samples was studied by X-ray line profile analysis. The X-ray line profiles were measured by a high-resolution rotating anode diffractometer (type: RA-MultiMax9, manufacturer: Rigaku) using $CuK\alpha_1$ ($\lambda = 0.15406$ nm) radiation. Two-dimensional imaging plates detected the Debye–Scherrer diffraction rings. The line profiles were determined as the intensity distribution perpendicular to the rings obtained by integrating the two dimensional intensity distribution along the rings. The line profiles were evaluated by the extended Convolutional Multiple Whole Profile (eCMWP) fitting procedure [29,30]. In this method, the diffraction pattern is fitted by the sum of a background spline and the convolution of the instrumental pattern and the theoretical line profiles related to the crystallite size, dislocations and twin faults. The details of the eCMWP fitting procedure can be found in Refs. [29,30]. Because of the ultrafine-grained microstructure of the studied samples, the physical broadening of the profiles was much higher than the instrumental broadening. Therefore, instrumental correction was not applied in the evaluation. The eCMWP method gives the area-weighted mean crystallite size ($\langle x \rangle_{area}$), the dislocation density (ρ) and the twin boundary probability (β) with good statistics, where the twin boundary probability is defined as the fraction of twin boundaries among the {111} lattice planes. The area-weighted mean crystallite size was calculated from the median (m) and the lognormal variance (σ^2) of the assumed lognormal crystallite size distribution as: $\langle x \rangle_{area} = m \cdot \exp(2.5\sigma^2)$. Additionally, a parameter denoted by q describing the edge/screw character of dislocations is also obtained from the eCMWP fitting.

EBSD investigations were carried out using a Zeiss Supra 40VP FEG scanning electron microscope (SEM). Depending on the size of the observed entities, the areas of scanned regions during EBSD experiments were adequately adapted using a step size between neighboring measurement positions from 50 to 250 nm. The average grain size, the fractions of low angle grain boundaries (LAGBs), high angle grain boundaries (HAGBs) and special Σ3 boundaries were extracted from the EBSD scans using OIM software version 5 from TexSem Laboratories (TSL).

2.4. In-situ transmission electron microscopy experiments

In-situ TEM was used to investigate the deformation mechanisms within the UFG component of the microstructure during tension. Specimens were prepared from the bulk material, first by cutting 3 mm × 1 mm × 0.5 mm rectangles that were eventually mechanically ground. The rectangles were then either thinned till perforation by electropolishing or by a combination of ion milling using Precision Ion Polishing System (PIPS™, Gatan, Inc., Model 691) and a "flash polishing" using an A2 electropolishing solution from Struers at a voltage of 7 V for 5 s at RT [31]. The latter sample preparation route was applied in order to remove the damaged surface layer caused by ion milling as a consequence of the faster etching rate of the UFG volumes compared to the CG ones during electropolishing. The samples were subsequently glued to a copper grid and placed into a Gatan straining holder. The in-situ TEM experiments were carried out in a JEOL 2010HC microscope operated at 200 kV. A MEGAVIEW III camera operating at a rate of 25 fps recorded the TEM images during deformation. The investigated areas were located on two sides of the hole in the perforated sample where the local stress is maximal [32].

To complement in-situ TEM observation, Automated Crystallographic Orientation Mapping (ACOM) in TEM was performed using the ASTAR technique [33]. The orientation maps were produced by a CM20FEG microscope operated at 200 kV.

3. Results and discussion

3.1. Microstructure of the starting powder and the sintered material

Fig. 1 shows a SEM micrograph of the initial Ni powder. The powder comprises spherical nanoparticles with the diameter between 50 and 100 nm in addition to ultrafine-grained and micrometer-sized particles (up to the size of 5 μm). Therefore, the initial powder has a bimodal particle structure.

After sintering, the microstructure of the as-consolidated sample was described as an ensemble of CG (the grain size is larger than 1 μm) and UFG (the grain size is smaller than 1 μm) volumes, i.e. as a bimodal microstructure. The as-processed bimodal-like microstructure is illustrated in Fig. 2, showing a map in which the various colors indicate the grains classified into different size ranges. The fractions of CG and UFG volumes computed from several EBSD images (Fig. 2 is only one of them) were found to be ~55 and ~45 vol.%, respectively. The average grain size in the UFG component of the microstructure was ~550 nm. In addition to quasi-random crystallographic texture (not shown), EBSD investigations showed that the CG entities can be subdivided into isolated micrometer-sized grains with an average grain size of ~2 μm and larger spherical multi-crystalline aggregates consisting of more than one coarse grain (see Figs. 2 and 3a). Actually, the isolated grains with an average grain size of ~2 μm and the spherical multi-crystalline clusters with the size up to ~30 μm occupy 51 and 4 vol.% of the whole microstructure, respectively. The average grain size within the multi-crystalline clusters is ~10 μm. Another characteristic feature of the microstructure is the high fraction of Σ3 CSL-type boundaries, appearing as red lines in both CG and UFG components in Fig. 3a. Such an