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Room-temperature deformation micro-mechanisms of polycrystalline nickel processed by spark plasma sintering

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ABSTRACT

The present work focuses on room temperature mechanical properties and deformation mechanisms of bulk polycrystalline nickel processed from a high purity micrometre-sized powder by means of spark plasma sintering. Optimisation of the conditions yielded bulk samples having a relative density ranging from 97 to about 99% and an average grain size in the range 5–45 μm. EBSD experiments were carried out to characterise the microstructure of as-processed sample before and after room temperature compression tests. The microstructure investigations prior to compression tests revealed a high density of high-angle grain boundaries (HAGBs); a large fraction of them are Σ3 boundaries (20 to 52% depending on the sample), the majority of which are twin boundaries (TBs). Compression tests (at a strain rate of 10^{-3} s^{-1}) at fixed amount of strains (5, 8 and 50%) result in an increase of the amount of low-angle grain boundaries, probably as a consequence of an intense dislocation activity. As for HAGBs, the more prominent effects occur for Σ3 boundaries and TBs for which a sharp decrease in the course of deformation is found. This was attributed to interaction with dislocations that possibly induce misorientation changes across boundaries (e.g., TBs) as well as their partial disruption as evidenced by EBSD investigations.

1. Introduction

Nickel (Ni) is a relatively abundant element found on the earth's crust and from which many metal alloys have been developed and used in various domains, such as aeronautics, medicine and defence. Indeed, Ni is often considered when a certain level of resistance to oxidation is needed. In addition, Ni possesses relatively good mechanical characteristics and therefore is an interesting structural material. As for many other materials, Ni-based structural parts can be advantageously produced by the versatile powder metallurgy (PM) route. Indeed, PM route offers many accessible parameters for processing bulk samples (pressure, temperature, holding times, etc.) [1]. These process parameters can be varied in order to get different microstructure types [2]. It is all the more relevant to control those parameters since the resulting microstructure itself has a decisive influence on mechanical properties in terms of strength and ductility especially [3–5]. These properties can be obtained only when the density is satisfactory and as close as possible to the theoretical density, but complete elimination of the porosity is a difficult challenge. PM processing includes several methods such as: Spark Plasma Sintering (SPS) [6], Hot Isostatic Pressing (HIP) [7,8], Cold Isostatic Pressing [9], Compaction and Sintering [10], Shock Consolidation [11], etc. Among the above-mentioned methods, SPS has the advantage to be run quickly. Contrary to the other PM methods, a few minutes are enough for the SPS densification mechanisms [1] to occur. In this technique, the sample is uniaxially pressed and subjected to few millisecond pulses of high electric current (up to thousands of amperes) that

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generates heat internally by Joule effect [12–14]. Actually, during SPS processing, the heating rate, the dwell time and temperature, and the applied pressure are important parameters. Temperature, which can be lowered due to the pressure assistance, does neither reach the melting point (1728 K for Ni) nor generate plasma state conditions [15] but is high enough for atomic diffusion and bonds between particles to occur. More details can be found in Ref. [16].

Compared to conventional methods, it has been shown that microstructures processed by SPS strongly depend on the sintering temperature [17]. Indeed, microstructural features such as grain size, the distribution of the grain boundary misorientation, and the coincidence site lattices (CSL) such as the Σ3 grain boundaries, evolve with the processing temperature. Therefore, all other parameters being fixed, the effect of the sintering temperature on the microstructure (along with the subsequent properties) is interesting to comprehend. In the present work, the influence of the temperature on the processed microstructure is analysed. The investigation additionally focuses on the mechanical properties under quasi-static compression tests at room temperature (RT) and the underlying deformation mechanisms for the most optimised sample (in terms of relative density and grain size) obtained here, are subsequently analysed and discussed.

2. Materials and Methods

2.1. *Nickel Powder*

The starting material was a high purity (99.9 wt.%) spherical Ni powder supplied by Sigma-Aldrich (St. Louis, MO, USA). A Scanning Electron Microscopy (SEM) micrograph shown in Fig. 1a illustrates the powder morphology; it consists of 1–5 μm multi-crystalline entities. Indeed, Fig. 1b represents a crystal orientation map showing the characteristic features of one particle after polishing of the initial powder and EBSD investigation. It shows that the particles are multi-crystalline and have no preferred orientation as illustrated by the EBSD map, where the colours represent poles in the ND direction (normal to the surface of observation) based on the [001] inverse pole figure colour key. Fig. 1c shows the positioning of the sample during EBSD investigations, where (ND, RD, TD) is an orthonormal basis attached to the sample. Notice that all the crystal orientation maps presented here are in accordance with the above description.

2.2. *Spark Plasma Sintering*

Powder densification was carried out by SPS using a 515S-SYNTEX machine at the regional SPS platform facility hosted by ICMPE (Thiais, France). With this setup, the maximum operating temperature is about 2273 K and the maximum pressure is about 120 MPa when a graphite die is used. The process takes place under a controlled argon atmosphere. In the present case, samples were consolidated under a 100 MPa uniaxial pressure and an electric direct current (1500 A maximum) subdivided into trains of 12 pulses separated by 2 pulses of 3.3 ms. Actually, considering instrumental limits, a uniaxial pressure of 50 MPa was first applied on the graphite die containing the powder. Temperature was then increased at a minimum rate of about 90 K min^{-1} while pressure increased up to 100 MPa. Sintering of the powder was performed at three dwell temperatures of 973 K, 1123 K and 1273 K. The corresponding samples are hereafter referred to N973, N1123 and N1273, respectively. The temperature and the maximum pressure of 100 MPa were applied during 1 min. The obtained samples are disc-shaped and have dimensions of about 20 mm in diameter and 4 mm in thickness.

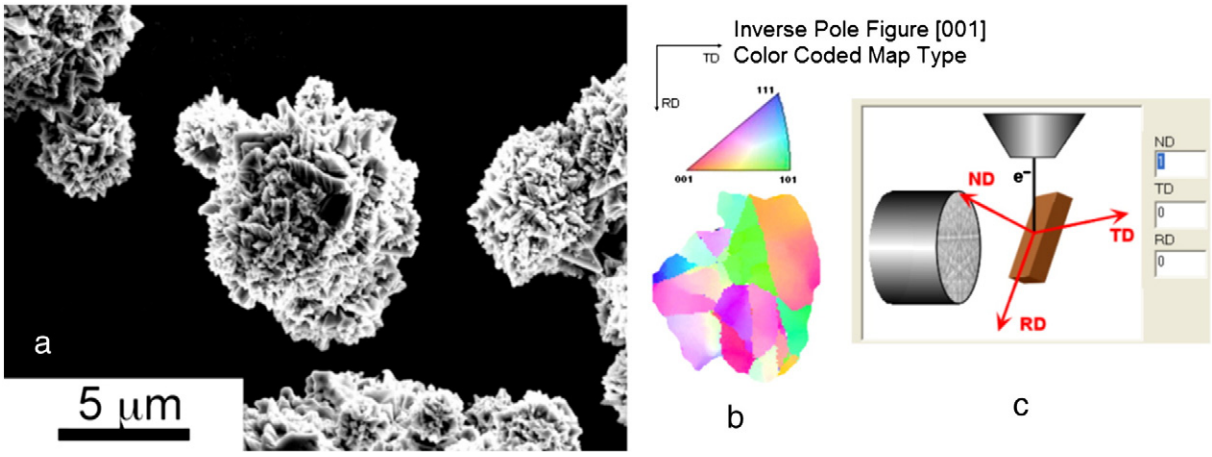

Fig. 1 – (a): SEM micrograph showing the morphology and structure of the starting high-purity (99.9 wt.%) Ni powder. (b): crystal orientation map showing the characteristic features of one particle after polishing of the initial powder and EBSD investigation. The colours represent poles in the ND direction (normal to the surface of observation) based on the [001] inverse pole figure colour key. (c): positioning of the sample during all the EBSD investigations where (ND, RD, TD) is an orthonormal basis attached to the sample.