Dense nanostructured nickel produced by SPS: Influence of the ball milling conditions on tensile properties.

Bolsonella Arnaud[1,3], Naimi Foad[2], Couque Hervé[3], Rémi Boulanger[3], Bernard Frédéric[1]

1. ICB – UMR 6303 CNRS / University de Bourgogne Franche-Comté, Dijon, France

2. SINTERmat – 7 rue alapetite, Montbard, France

3. Nexter Munitions – 7 route de Guerry, Bourges, France

Highlights

- Contamination of nickel powder by high-energy milling
- Sintering of nanostructured nickel powders with optimal properties
- Relation between microstructure and mechanical properties of milled powder and dense Ni.

1. Introduction

The aim of the present research work was to fabricate dense nanostructured nickel in order to take advantage of the improvement of mechanical properties with reduced grain size. One of the challenges was to limit the grain coarsening even in significant size specimens, through the control of the SPS processing parameters (such as heating rate, sintering temperature, holding time, and pressure). However, it is also crucial to control the production of nanopowders in order to get powders having a nanometric size and a size distribution as narrow as possible. Such a control is not obvious due to the large reactivity of nanoparticles. That is why as-milled powders have been produced using high-energy ball milling. Indeed, the milling process leads to both the reduction in the crystallite size and the accumulation of defects in powder particles. The other interest of ball milling is the possibility to form agglomerates with a micrometer size (size usually available with commercial ones) containing nano-crystallites. However, ball milling conditions have also an influence on the purity of milled powders by a possible contamination from milling tools. Therefore, the objective of this work is to study the influence of the microstructure (crystallite size and crystallite deformation rate) and the purity (milling contamination) of the Ni milled powders on the mechanical properties of Ni sintered by SPS.

2. Methods

The process to produce dense nanostructured Ni from a micrometric commercial powder consists in two steps: (i) mechanical activation of the elemental powder by a short-duration treatment in a high-energy planetary milling (milled powder) and (ii) densification of Ni in one step by SPS.
The SPS samples were 60 mm in diameter and 10 mm in height allowing the extraction of three tensile specimens. Powder preparation was realized as to enable one SPS sample with 230 g of elemental powder Ni milled in a planetary ball vario-mill Fritsch Pulverisette 4. A fine description of these powders in terms of purity (surface and bulk), crystallite size and strain rate was investigated using SEM-EDX, XPS, TGA, and XRD analysis respectively. The tensile tests were performed at a strain rate of $10^{-3}s^{-1}$ with specimens 16 mm in gage length and 4 mm in gage diameter using a Testwell machine. The load was measured with a load cell of capacity 5000 daN. The elongation was measured with an extensometer Epsilon over a length of 10 mm.

3. Results and discussion

Based on previous works [1], a specific ball milling condition was established at 250 rpm (rotation per minute) for the disk rotation speed and −50 rpm for the absolute vial rotation speed. The charge ratio (steel ball to powder mass ratio) was 7. It was decided to study the influence of the milling duration (1h to 8h) on the crystallite size and the strain rate. In parallel, the possible contamination by the milling tools and the oxygen initially present at the surface of commercial powders is also followed. As expected, the crystallite size (from 110 nm to 70 nm) decreases as a function of milling time while, at the same time, the deformation rate increases (from 32% to 41%).

Additionally, TGA analyzes highlight two mass losses (starting from 185°C and 685°C) led to the milling process performed during 8h as shown on Figure 1.

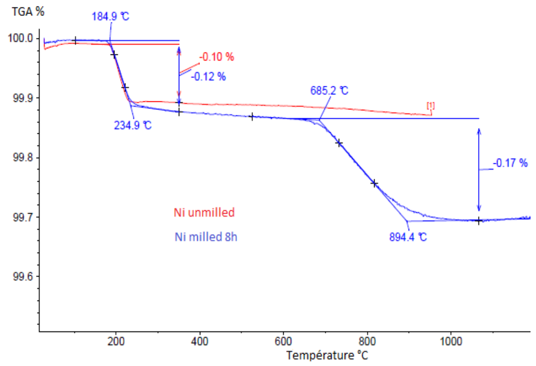

Figure 1. TGA for unmilled (red) and milled (blue) powders.

These mass losses are compared with those obtained on the commercial powder (without milling) which has only a single mass loss at 185°C. As well, these mass losses were able to be attributed respectively to the presence of oxide layer on the surface of particles (1) and oxygen trapped inside agglomerates/aggregates (2). Finally, after limited to a maximum these contaminations, sintering by SPS of these powders has been able to be carried out in order to obtain dense materials. As indicated in previous work, WC-CO tools have been used in order to apply an axial pressure close to 200 MPa and thus to reach a density close to 100%. The tensile curves versus milling conditions were established (Figure 2).

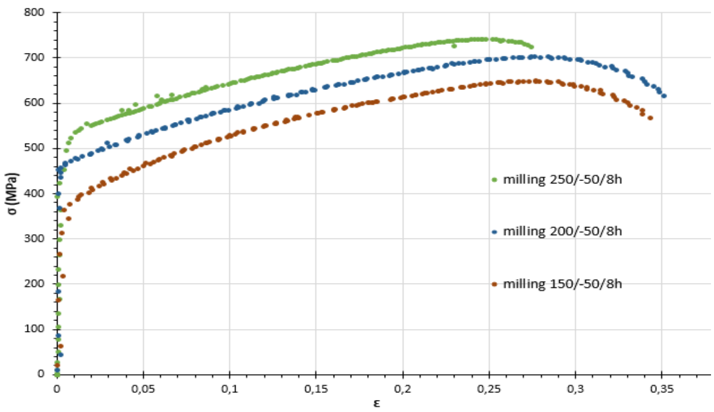

Figure 2. True tensile stress-strain curves of SPS samples elaborated by different Ni milled powder.

These curves reveal that the mechanical properties of the dense Ni sintered from mechanically activated powders (i.e. nanostructured) are far superior to that obtained with Ni electrodeposited (σ_V = 220 MPa, ε = 42% [1]). Such results are more promising since it possible to modulate the couple stress / strain by a control of the milling conditions.

4. Conclusions

High strength nickels with significant ductility were obtained with the SPS process. These results provide a procedure to identify the “SPS window” for the elaboration of nickels with a controlled microstructure and purity.

References

[1] F. Naimi, L. Minier, H. Couque, F. Bernard, Journal of Nanomaterials, Volume 2013 (2013), Article ID 674843

Acknowledgments

Support of the Délégation Générale de l’Armement under contracts N°201392090003 et N°ANRT/CIFRE-Défense 0003/2016/DGA is gratefully acknowledged.