Production of high-strength Al$_{85}$Y$_8$Ni$_5$Co$_2$ bulk alloy by spark plasma sintering

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Abstract. Highly dense bulk samples were produced by spark plasma sintering (SPS) through combined devitrification and consolidation of partially amorphous Al$_{85}$Y$_8$Ni$_5$Co$_2$ gas atomized powders. The microstructure of the consolidated samples shows a mixed structure containing crystalline, ultrafine-grained and amorphous/nanocrystalline particles. The sintered sample exhibits a remarkable high strength of about 1050 MPa combined with 3.7 % fracture strain.

1. Introduction

In recent years, significant efforts have been dedicated to the production of amorphous, partially amorphous, nanocrystalline and ultra-fine grained (UFG) Al-based alloys [1-5]. Due to their high strength combined with low density, these alloys are very attractive candidates for structural and functional applications [1-5]. For example, Al-based amorphous alloys with more than 85 at.% Al, such as Al$_{85}$Y$_8$Ni$_5$Co$_2$, show a tensile fracture strength exceeding 1200 MPa combined with good bending ductility [6]. Partially crystallized Al-based amorphous ribbons with a microstructure consisting of an uniform dispersion of fcc Al nanoparticles within the residual amorphous matrix exhibit even higher fracture strengths and hardness with respect to fully amorphous alloys of the same composition [7-11]. However, in spite of their promising mechanical properties, the maximum scale of these materials is generally limited to a thickness of less than 100 micrometers, which strongly restricts their engineering application.

To overcome this limitation, powder metallurgical methods, including powder consolidation, have been used to produce bulk high strength Al-based materials [4]. Among the consolidation techniques used, spark plasma sintering (SPS) is particularly attractive due to many advantages, including good densification, a rapid consolidation process and reduced grain growth [12-13]. Accordingly, in this
work, partially amorphous Al$_{85}$Y$_8$Ni$_5$Co$_2$ gas atomized powders were consolidated by SPS. Consolidation by SPS results in a heterogeneous microstructure consisting of crystalline, nanocrystalline and amorphous phases, which leads to promising room temperature mechanical properties, i.e. high strength combined with good plastic deformation.

2. Experimental Details
Gas atomized partially amorphous powder with nominal composition of Al$_{85}$Y$_8$Ni$_5$Co$_2$ (at.%) were used as starting material. The Phase formation and the microstructure were studied by X-ray diffraction (XRD) using a Philips PW 1050 diffractometer (Co K$_\alpha$ radiation) and by scanning electron microscopy (SEM) using a JEOL JSM 6400 microscope. The thermal stability of the as-atomized powders was studied by differential scanning calorimetry (DSC) at a heating rate of 40 K/min with a Perkin-Elmer DSC7 under a continuous flow of purified argon. The powders were consolidated into cylindrical specimens of 10 mm diameter under high vacuum by spark plasma sintering at a temperature of 673 K (heating rate of 10 K/min) and an applied pressure of 500 MPa using a Sumitomo SPS-515S sintering machine. The density of the consolidated samples was evaluated by the Archimedes principle and was found to be 99 \% of the theoretical density. Cylindrical specimens with a length/diameter ratio of 2 (6 mm length and 3 mm diameter) were prepared from the SPS samples by wire erosion method. The specimens were tested with an INSTRON 8562 testing facility under quasi-static compressive loading (strain rate of 1x10$^{-4}$ s$^{-1}$) at room temperature. Both ends of the specimens were polished to make them parallel to each other prior to the compression test.

3. Results and discussion
Figure 1 displays the XRD pattern of the as-atomized Al$_{85}$Y$_8$Ni$_5$Co$_2$ powder. The pattern shows a weak broad maximum characteristic of an amorphous structure centered at about 45° along with the diffraction peaks from fcc Al, and Al$_3$Y and Al$_6$Co$_2$ intermetallic phases. This indicates that the cooling rate during gas atomization is not high enough to suppress the formation of crystalline phases [14]. Figure 2 shows the isochronal DSC scan (40 K/min) of the as-atomized powder and the shrinkage rate behavior during the SPS process. The DSC scan reveals a clear glass transition ($T_g$) at 550 K followed by three exothermic peaks with onset temperatures $T_{x1} = 570$ K, $T_{x2} = 610$ K and $T_{x3} = 660$ K due to crystallization of the glassy phase. A similar multi-step crystallization behavior has been observed by several authors [6,15-18] and corresponds to the formation of fcc Al in the first crystallization event followed by the formation of the Al$_3$Y and Al$_6$Co$_2$ phases at higher temperatures [6,16-17].

During consolidation by SPS a constant pressure of 500 MPa was applied from room temperature through the crystallization events to the final sintering temperature (673 K to avoid complete

Figure 1. XRD patterns of the as-atomized powder and the bulk sample consolidated by SPS at 673 K.

Figure 2. Isochronal DSC scan (40 K/min) of as-atomized powder and shrinkage rate of the powder during SPS (data points).
crystallization), which was held for about 3 minutes. The consolidation of the Al_{85}Y_{8}Ni_{5}Co_{2} powder at high temperatures can thus be considered as a combined in-situ (partial) devitrification and densification of the powder. The shrinkage rate during SPS (data points in Figure 2) reveals two maxima at about 550 and 670 K, respectively, which are associated with the highest densification rates. These two main shrinkage events take place at the glass transition region (where the glassy phase transforms into the supercooled liquid) and in the temperature range between the second and the third crystallization events, where the residual amorphous phase with a different composition may undergo the glass transition, as observed for the Al_{87}Ni_{8}La_{5} glassy powder [19].

![Figure 3. SEM micrographs of the sample sintered at 673 K.](image)

![Figure 4. Room temperature true stress-true strain curve of the consolidated sample.](image)

The XRD pattern of the Al_{85}Y_{8}Ni_{5}Co_{2} powder sintered at 673 K is also shown in Figure 1. The structure is similar to the as-atomized powder and consists of fcc-Al together with the two intermetallic compounds Al_{3}Y and Al_{9}Co_{2}. The pattern still shows a weak broad maximum at about 45°, indicating that an amorphous phase is retained after sintering. The diffraction peaks in Figure 1 are rather broad, indicating that the phases formed are of nano sized or ultra-fine dimensions.

Figure 3 shows SEM images of the sample sintered by SPS at 673 K. The microstructure is rather heterogeneous, consisting of particles with different characteristics: small featureless particles (most likely with nano-crystalline or amorphous structure), particles with large crystalline features, ultrafine-grain structured particles and particles with mixed crystalline/nano-crystalline/amorphous features. EDX investigations reveal that the dark areas between the particles in Figure 3 correspond to fcc Al.

A typical room temperature uni-axial compression stress-strain curve for the sintered sample is shown in Figure 4. The material exhibits a yield strength (0.2 % offset) of about 800 MPa followed by a region with strain hardening up to the maximum stress of 1050 MPa and fracture strain of 3.7 %.

Spark plasma sintering of the Al_{85}Y_{8}Ni_{5}Co_{2} powder leads to highly dense specimens displaying high strength combined with good plastic deformation. Such a behavior is presumably due to the multi-phase microstructure consisting of soft fcc-Al and high-strength intermetallic compounds (see Figures 1 and 3). However, the observed room temperature plastic deformation is in contrast to what was reported for other Al-based alloys produced by consolidation of gas-atomized powders [4], which display a similar microstructure. For example, although fully crystallized Al-Ni-Y-Co samples exhibit an extremely high compressive strength of 1420 MPa [4], the plastic strain is only about 1 % [20]. This is similar to recent results of Sasaki et al. [21] on nanocrystalline Al_{85}Ni_{10}La_{5} samples produced...
by SPS of gas-atomized amorphous powders, which show a compressive strength exceeding 1200 MPa, but no plastic deformation. A possible explanation for the larger plastic deformation of the present material may be linked to the heterogeneous microstructure characterizing the consolidated sample, consisting of crystalline, nanocrystalline and amorphous phases (Figure 3), which might provide the right balance between soft and hard phases explaining the observed high strength combined with good plastic deformation.

4. Summary

In-situ devitrification and consolidation of gas-atomized Al$_8$Y$_8$Ni$_5$Co$_2$ powders into highly dense bulk specimens was carried out by spark plasma sintering. Room temperature compression tests of the consolidated bulk material reveal remarkable mechanical properties, namely, a high compression strength of 1050 MPa combined with a fracture strain of about 3.7%. These findings demonstrate that the combined devitrification and consolidation of glassy precursors by spark plasma sintering is a suitable method for the production of Al-based materials characterized by high strength and good plastic deformability.

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