Arch. Metall. Mater. **66** (2021), 4, 1055-1058

DOI: 10.24425/amm.2021.136424

JONGMIN BYUN[1], YOUNG-IN LEE[1], SUNG-TAG OH[1*]

HOMOGENEOUS DISPERSION OF YTTRIUM OXIDE PARTICLES IN NICKEL-BASED SUPERALLOY BY HIGH PRESSURE HOMOGENIZING AND BALL MILLING METHOD

An optimum route to fabricate the Ni-based superalloy with homogeneous dispersion of Y_2O_3 particles is investigated. Ni-based ODS powder was prepared by high-energy ball milling of gas-atomized alloy powders and Y_2O_3 particles treated with a high-pressure homogenizer. Decrease in particle size and improvement of dispersion stability were observed by high-pressure homogenization of as-received Y_2O_3 particles, presumably by the powerful cavitation forces and by collisions of the particles. Microstructural analysis for the ball-milled powder mixtures reveal that Ni-based ODS powders prepared from high-pressure homogenization of Y_2O_3 particles exhibited more fine and uniform distribution of Ni and Y elements compared to the as-received powder. These results suggested that high-pressure homogenization process is useful for producing Ni-based superalloy with homogeneously dispersed oxide particles.

Keywords: Ni-based superalloy, Y_2O_3 dispersion, High pressure homogenizer, Spark plasma sintering, Microstructure

1. Introduction

Ni-based superalloys are promising materials for gas turbines in advanced power plant and aerospace applications due to their excellent high temperature strength and resistance to degradation in corrosive or oxidizing environments [1,2]. Their remarkable mechanical performance is normally achieved by solid solution strengthening of the γ matrix phase, precipitation strengthening of γ′ precipitates and oxide dispersion strengthening (ODS) using the homogeneous dispersion of stable oxide nanoparticles into the matrix [3-5]. Generally, processing of Ni-based ODS superalloys involves mechanical alloying (MA) of powders, followed by pressure-assisted sintering. The MA process is the core step to manufacture ODS superalloys, involving that the elemental alloying powders or oxide compounds are subjected to the high energy ball milling to allow the oxide nanoparticles to be dispersed homogeneously throughout the matrix powders [6,7].

However, nanometer-scale refining and homogeneous dispersion of micro-sized oxide particles in relatively ductile matrix is not easily achieved in MA processes, requiring long milling times. In order to solve this problem, various processes have been developed including MA process using the nano-sized oxides as starting powders instead of micro-sized oxides, internal oxidation method, sol-gel and polymeric additive solution routes [8-10]. Also, a process using a high pressure homogenizer has been reported as a promising approach for removing aggregates of the nanopowder [11]. This route is typically performed by forcing a liquid through a narrow nozzle at high pressure and by such establishing high shear stress. Thus, it is possible to prepare a suspension with monodisperse particles from which aggregates have been removed through high pressure homogenization.

The aim of the present work is to investigate high pressure homogenization route as a potential method to fabricate the Ni-based superalloys with homogeneously dispersed Y_2O_3 nanoparticles. Ni-based ODS powders were prepared by high-energy ball milling of gas-atomized alloy powders and Y_2O_3 particles treated with a high-pressure homogenizer, and then were consolidated by spark plasma sintering. The microstructure and distribution of oxide nanoparticles were characterized.

2. Experimental

The superalloy powder having a nominal composition of Ni-15Cr-4.5Al-4W-2.5Ti-2Mo-2Ta-0.15Zr in wt% was manufactured by gas atomization. The atomized powder was supplied by Korea Sintered Metal Company. Two different fabrication processes were applied to obtain the Ni-based ODS powder

[1] SEOUL NATIONAL UNIVERSITY OF SCIENCE AND TECHNOLOGY, DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING & THE INSTITUTE OF POWDER TECHNOLOGY, SEOUL 01811, REPUBLIC OF KOREA

* Corresponding author: stoh@seoultech.ac.kr

mixtures. The first one used atomized powder and as-received Y_2O_3 (<50 nm, Sigma-Aldrich Co., USA) of 1.1 wt% in the final ODS powder mixture. The powders were high-energy ball milled for 15 h using horizontal ball milling machine (Simoloyer, Zoz GmbH, Germany) with a ball-to-powder weight ratio of 15:1. In the second method, Y_2O_3 particles treated with a high-pressure homogenizer was used. The raw Y_2O_3 powder was mixed in ethanol for 3 min using acoustic mixer (PharmaRAM™ I, Resodyn Acoustic Mixers Inc.). The slurry was passed through a high-pressure homogenizer (Panada Plus 2000, GEA, Germany) with a micro orifice module operated at 1000 bar for 3 cycles. The atomized powder was then mixed with the high-pressure homogenized Y_2O_3 solution and wet ball-milled for 3 h. The prepared mixtures were completely dried in air for 24 h and high-energy ball-milled for 15 h.

The superalloy powders were sintered at 1000°C for 10 min in vacuum under a pressure of 30 MPa using spark plasma sintering (SPS, Sumitomo Coal Mining Co.). The size of prepared powders was measured by dynamic light-scattering method (DLS-8000, Otsuka Electronics, Japan). The dispersion stability was measured by detecting concentration variation in the suspension by scanning the whole height of the sample in backscattering, using a Turbiscan (Formulaction, France) [12]. The microstructure and elemental mapping were observed by scanning electron microscopy (SEM, JSM-6700F, JEOL Co., Japan) equipped with an electron probe micro-analyzer (EPMA).

3. Results and discussion

Typical morphology and particle size distribution of as-received and high-pressure homogenized Y_2O_3 particle are shown in Fig. 1(a) and (b), respectively. In the initial powder (Fig. 1(a)), the large agglomerates with the mean particle size of 201 nm were observed. Conversely, as shown in Fig. 1(b), the agglomerates are partially removed by the high-pressure homogenization, and

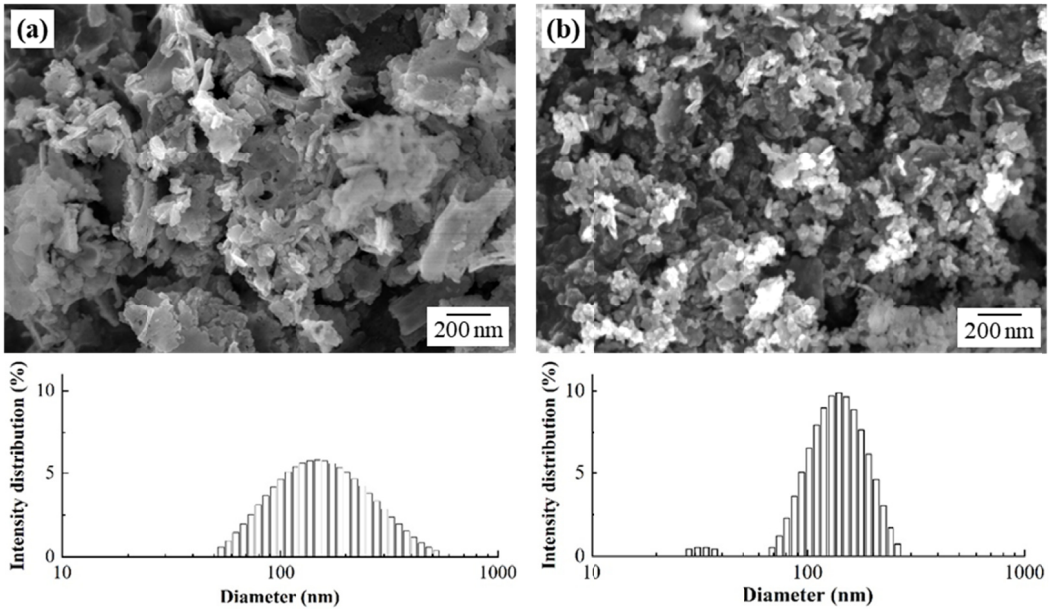

Fig. 1. Micrographs and particle size distribution of (a) as-received and (b) high-pressure homogenized Y_2O_3 particles

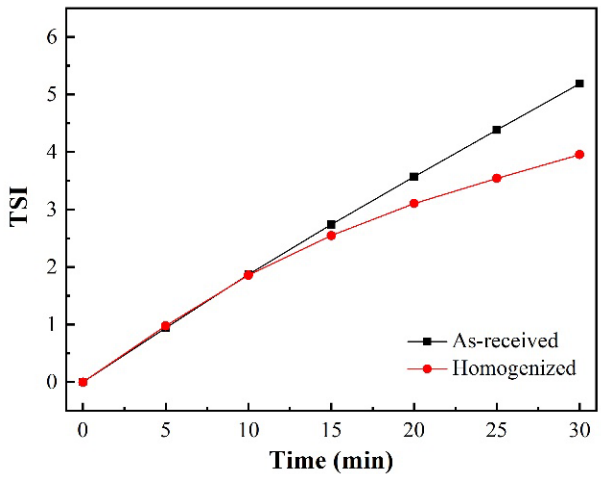

Fig. 2. TSI value of the Y_2O_3 suspension before and after homogenization

Y_2O_3 with mean size of 139.6 nm exist in the form of fine particles. The high-pressure homogenizer is essentially a bottleneck through which the suspension passes with a high velocity, thus experiencing a significant pressure drop, turbulent flow conditions and cavitation phenomena [13]. Therefore, it is clear that the high-pressure homogenization method significantly comminuted Y_2O_3 aggregates, presumably by the powerful cavitation forces arising from the gas bubbles and by collisions of the particles with each other and with the homogenizer.

For the analysis of the effect of high-pressure homogenization on dispersion stability, Turbiscan stability index (TSI) was calculated based on backscattering changes in Turbiscan [14]. As shown in Fig. 2, the TSI of the homogenized suspension showed a relatively low value, that is, enhanced stability compared to the raw powder. From consideration of the measured

particle size and TSI value, the high-pressure homogenization is expected to contribute to the homogeneous dispersion of Y_2O_3 particles in the ball milling process by enabling the comminution of the agglomerates and improvement of the dispersion stability.

The typical morphologies of atomized powder and ball-milled mixture are shown in Fig. 3. The atomized powder is spherical, and the mean particle size was measured as 84.5 μm. Fig. 3(b) and (c) show SEM images of Ni-based ODS powders prepared from ball milling using as-received and high-pressure homogenized Y_2O_3 powders, respectively. In the case of ball milling of atomized powder and as-received Y_2O_3, relatively large particles with the mean size of 54.3 μm were observed, while the powder prepared using high-pressure homogenized Y_2O_3 powders exhibited finer particle size as 30.2 μm. This difference in powder size could be explained by comminuted agglomeration and improved dispersion stability effect of homogenized Y_2O_3 powders in the ball milling process [15].

In order to characterize the microstructure of ball-milled powders, the distribution of alloy elements is analyzed by SEM and EPMA mapping, as shown in Fig. 4. Agglomerated

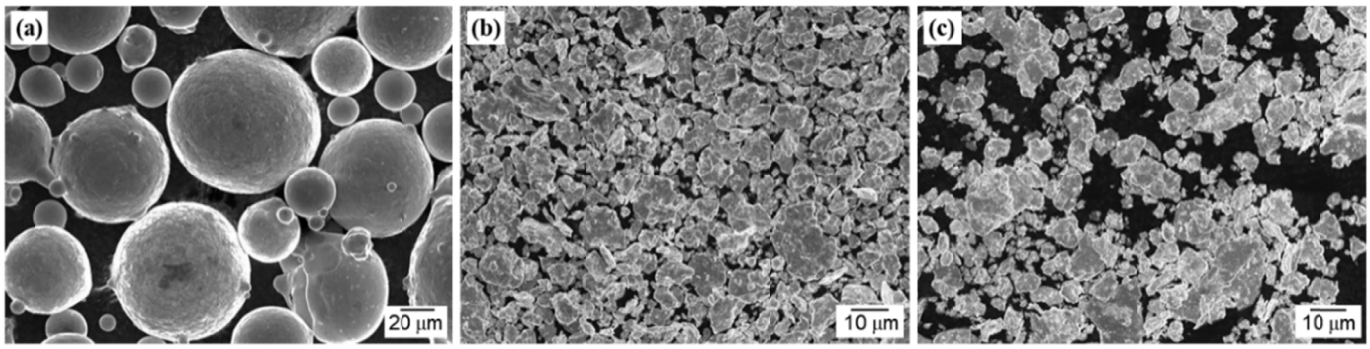

Fig. 3. SEM morphologies of (a) gas-atomized powder; Ni-based superalloy powder fabricated by high-energy ball milling of atomized powder mixed with (b) raw and (c) high-pressure homogenized Y_2O_3 particles

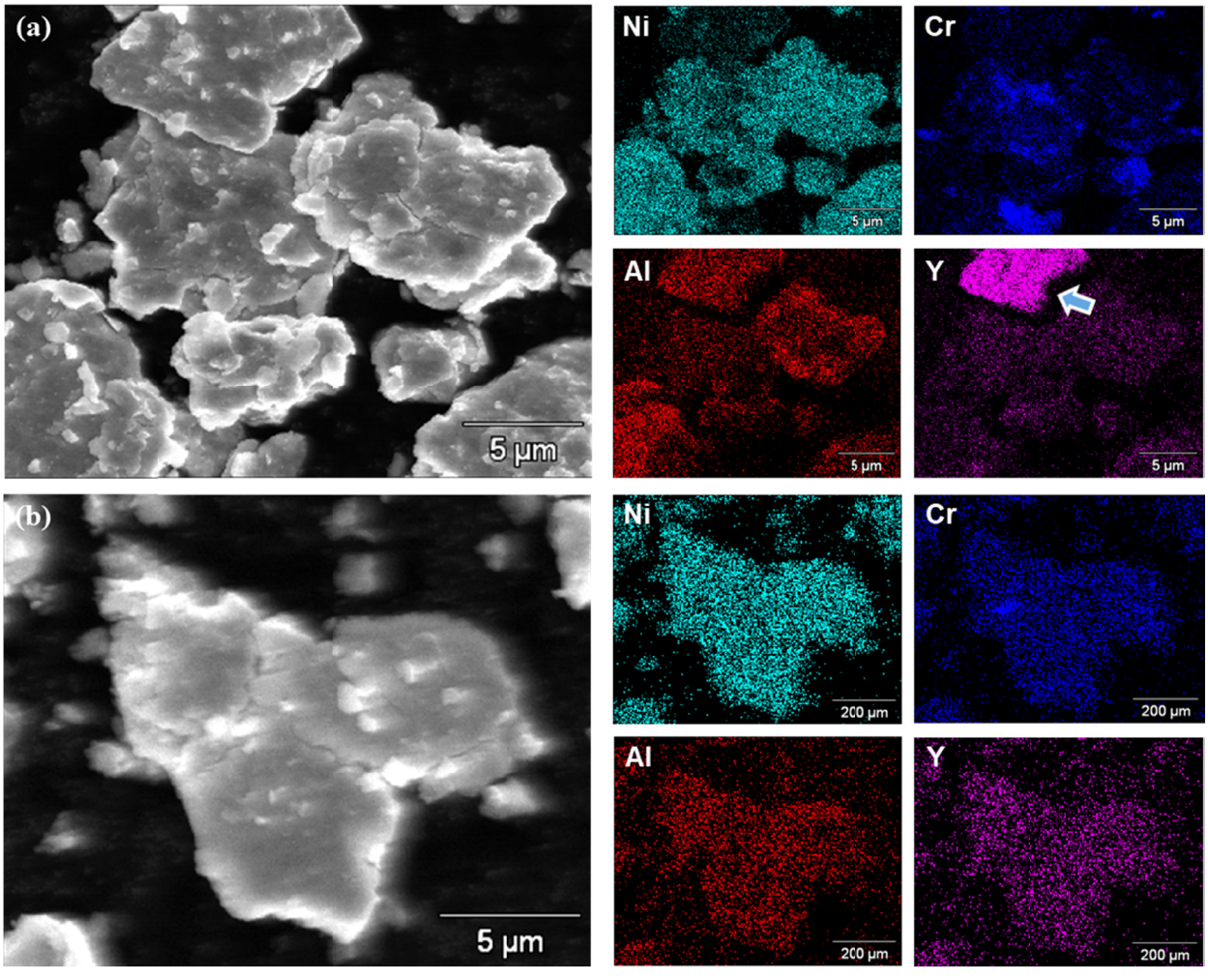

Fig. 4. SEM-EDX analysis of powder mixture, ball-milled by using (a) raw and (b) high-pressure homogenized Y_2O_3 particles

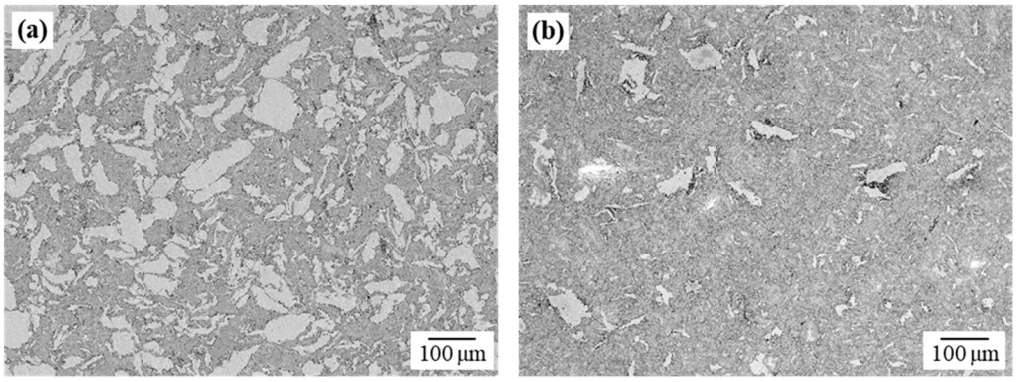

Fig. 5. SEM images of the microstructure after spark plasma sintering of powder mixture using (a) as-received and (b) high-pressure homogenized Y_2O_3 particles

Y element indicated by arrows were observed in the ball-milled powder using as-received Y_2O_3, as shown in Fig. 4(a). However, it clearly represented a more uniform distribution of Y in the powder mixture prepared from high-pressure homogenized Y_2O_3 powders (Fig. 4(b)). From this observation, it is suggested that high-pressure homogenization process is useful for producing Ni-based superalloy powders with homogeneously dispersed oxide particles.

The synthesized powders were sintered at 1000°C for 10 min in vacuum under a pressure of 30 MPa to make consolidated ODS alloys. As clearly seen from Fig. 5(a), the sintered specimen prepared from ball-milled powder using as-received Y_2O_3 exhibited inhomogeneous and large grains. However, the specimen using high-pressure homogenized Y_2O_3 powders showed a relatively fine microstructure due to uniform dispersion of fine oxide particles in powder mixture. Therefore, the high-pressure homogenization process is one of useful approaches to fabricate the Ni-based ODS superalloy with homogenous dispersion of Y_2O_3 nanoparticles.

4. Conclusions

The present study has focused on fabrication and dispersion control of Y_2O_3 particles by using a ball milling and high-pressure homogenization. Two methods for the preparation of Ni-based ODS powders have been presented and discussed on the basis of microstructural characteristics. The powder mixture fabricated by the ball milling using atomized Ni-base powder and homogenized Y_2O_3 particles showed homogeneous distribution of oxide particles compared with that using as-received Y_2O_3 particles, presumably by the cavitation forces and by collisions of the particles. This result indicated that nanoparticle powders with controlled dispersion can be obtained by ball milling and high-pressure homogenization.

Acknowledgments

This study was supported by the Research Program funded by the SeoulTech (Seoul National University of Science and Technology).

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