Oxide reinforced Ni base composite prepared by spark plasma sintering

Guoliang Zhu[1,2,a], Shiqi Zhao[3], Rui Wang[1,2], Anping Dong[1,2], Liang Zhang[4,5], Wenheng Wu[4,5], Wei Wang[1,2], Sheng Jiang[6] and Yilong Pu[6]

[1]*School of Materials Science and Engineering, Shanghai Jiao Tong University, Shanghai 200240, China*
[2]*Shanghai Key Laboratory of Advanced High-temperature Materials and Precision Forming, Shanghai 200240, China*
[3]*School of Mechanical Engineering, Shanghai Jiao Tong University, Shanghai 200240, China*
[4]*Shanghai Engineering Research Center of 3D Printing Materials, Shanghai 200437, China*
[5]*Shanghai Research Institute of Materials, Shanghai 200437, China*
[6]*Jiangsu Longda Superalloy Material Co., Ltd, Wuxi 214105, China*

Abstract. Y_2O_3 is a favourable reinforcement for the oxide dispersion strengthened superalloy due to its good high temperature stability, excellent strength and large elastic modulus. In this work, Y_2O_3 is introduced into the K4169 superalloy aiming to improve its high temperature properties. The high quality Y_2O_3 and K4169 powder mixture can be obtained with the addition of the process control agent of stearic acid after high energy ball milling, and the Y_2O_3/K4169 composite with uniformly dispersed reinforcement of nanoscale Y_2O_3 particles are finally synthesized with the ball-milled powders by using spark plasma sintering.

Introduction

Different from the technical route of the optimization of composition and microstructure, the oxide dispersion strengthening (ODS) is an effective method to improve the high temperature performances of materials [1]. Thus, the synthesis of ODS superalloys has attracted wide attention. Yttria (Y_2O_3) is a common reinforcement for ODS superalloy due to its high thermal stability, favourable strength and elastic moudulus [2]. Benjamin [3] has prepared the Y_2O_3 reinforced superalloy by using mechanical alloying in 1970s. Later, a few ODS superalloys have been invented, including MA754 and PM3000, in which the concentration of Y_2O_3 is 0.6wt.% and 0.9wt.% Y_2O_3 respectively. The superalloys strengthened by Y_2O_3 have been widely applied in aero engines. Moreover, Y_2O_3 also can react with the elements in matrix or other added elements to form new reinforcing phases, resulting in better strengthening effect. The $Y_3Al_5O_{12}$, $YAlO_3$ and $Y_4Al_2O_9$ phases are formed in the MA6000 alloy due to the reactions between Y_2O_3 and Al during the mechanical alloying [4]. the addition of Hf in PM1000 ODS superalloy leads to the formation of $Y_2Hf_2O_7$ which has better interface characteristics with the matrix compared to Y_2O_3 [5].

K4169 alloy is a commercial superalloy which has similar composition and microstructure with IN718C alloy. The high temperature strength of K4169 is not satisfactory though it has excellent comprehensive properties when the service temperature is lower than 650ºC [6-8].

In this work, Y_2O_3/K4169 composite is prepared by using high energy ball milling (HEBM) and spark plasma sintering (SPS) to improve the high temperature strength of matrix K4169 superalloy. The microstructures of the ball-milled Y_2O_3 and K4169 powders as well as the microstructure of Y_2O_3/K4169 composite after SPS are studied.

[a] Corresponding author: glzhu@sjtu.edu.cn

1 Materials and method

High-purity Y_2O_3 powders and K4169 powders are used as raw materials. Spherical K4169 powders (45 μm) and nanoscale Y_2O_3 powders (50 nm) are added to a ball milling jar. The weight ratio of Y_2O_3 in the powder mixture is 10wt.%, and 0.5wt.% of stearic acid is used as process control agent for HEBM. The weight ratio of steel balls to the powder mixture is 10:1. The HEBM is conducted with a rotating speed of 300 rmp, and the ball milling time is 12h. After HEBM, the passivation of powders is conducted under 0.2 atm for 30 min in an argon glove box to avoid self-burning due to the high activity of ball-milled powders. Then the ball-milled powder mixture is sintered in SPS apparatus at 1050 ºC under the pressure of 50 MPa for 5 min.

The microstructure of ball-milled powders before SPS and the Y_2O_3/K4169 composite after SPS are investigated by using scanning electron microscope (JSM7600F). The phases of the ball-milled powders before SPS and the Y_2O_3/K4169 composite after SPS are analysed by X-ray diffractometer (Bruker AXS D8), Cu target and a scanning speed of 0.2 °/s are employed for XRD tests.

2 Results and discussion

Figure 1 shows the SEM images of the ball-milled powders. The size of the K4169 powders is greatly decreased from 45μm to 20μm. No obviously scattered Y_2O_3 particles are found in the ball-milled powders, indicating that the nanoscale Y_2O_3 powder and microscale K4169 powders are homogeneously mixed after HEBM.

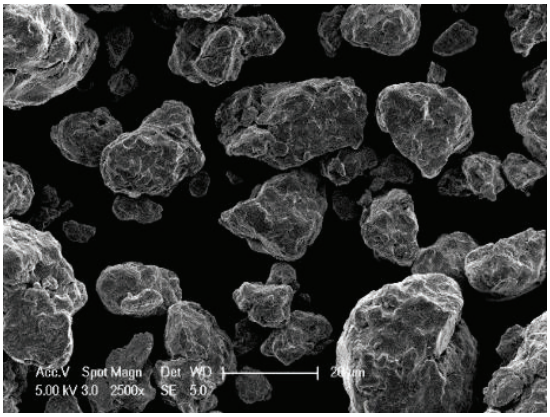

Figure 1. SEM morphology of the ball-milled Y_2O_3 and K4169 powders after HEBM.

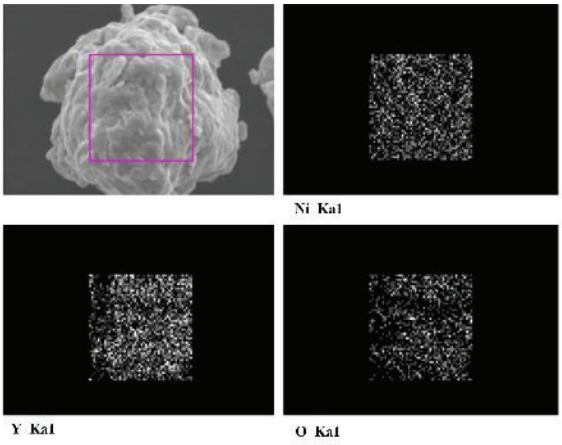

Figure 2. The distribution of elements in the ball-milled Y_2O_3 and K4169 powders after HEBM.

The energy dispersive spectrometry results of a ball-milled powder, as shown in Figure 2. Both oxygen and yttrium are uniformly distributed in the ball-milled powder, without obvious aggregation. The result confirms that high quality Y_2O_3 and K4169 powder mixture can be prepared after HEBM for 12h with the help of process control agent of stearic acid.

Figure 3 shows the XRD pattern of the ball-milled Y_2O_3 and K4169 powders after HEBM. The characteristic diffraction peaks of Ni and Y_2O_3 are clear and sharp. Moreover, no other characteristic diffraction peak is found in Figure 3, indicating that no impurity is formed during the HEBM.

The XRD pattern of Y_2O_3/K4169 composite after SPS is shown in Figure 4. The characteristic diffraction peaks of γ phase and Y_2O_3 are observed, indicating that the Y_2O_3 was thermal stable during the SPS. Considering that the standard heat treatment is not carried out for Y_2O_3/K4169 composite after SPS, it is reasonable that the characteristic diffraction peaks of γ′ and γ″ phases are not found in the XRD pattern.

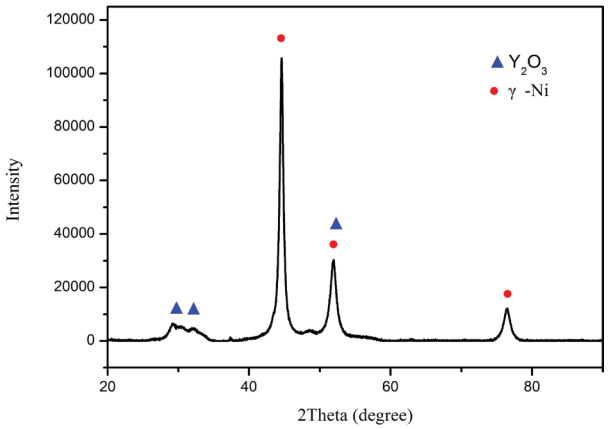

Figure 3. XRD pattern of the ball-milled Y_2O_3 and K4169 powders after HEBM.

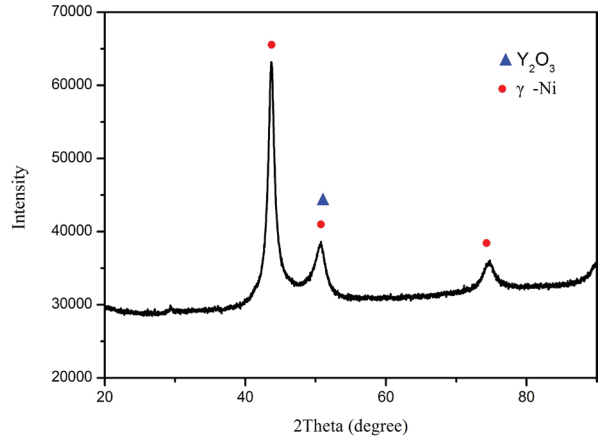

Figure 4. XRD pattern of Y_2O_3/K4169 composite after SPS.

It is believed that the nanoscale Y_2O_3 could enhance the material strength through the dislocation pinning mechanism. With the coarsening of γ′ and γ″ phases during long time service of Y_2O_3/K4169 composite, the strengthening effect from γ′ and γ″ is greatly decreased, but the Y_2O_3 could still work as reinforcement to strengthen the matrix, due to its high thermal stability. In the future, the deformation behavior and the microstructure evolution after heat treatment will be investigated to further optimize the microstructure of Y_2O_3/K4169 composite, aim to improve the high-temperature performance of Y_2O_3/K4169 composite.

3 Conclusion

The high quality Y_2O_3 and K4169 powder mixture can be obtained by HEBM which is performed with a rotational speed of 300 rmp for 12 h under argon protection, and the Y_2O_3/K4169 composite with uniformly distributed nanoscale Y_2O_3 particles is prepared by SPS of the ball-milled powders. It is believed that the nanoscale Y_2O_3 in Y_2O_3/K4169 composite could greatly enhance the material strength through the dislocation pinning mechanism during long time service due to its high thermal stability. The optimization of the microstructure of Y_2O_3/K4169 composite will be further investigated in the next research work.

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