MICROSTRUCTURE AND THERMAL STRESS RELAXATION OF ZrO_2-Ni FUNCTIONALLY GRADED MATERIAL ①

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ABSTRACT The microstructural characteristics and thermal stress relaxation of ZrO_2-Ni functionally gradient materials (FGM) by hot-pressing were studied. The investigation by scanning and transmission electron microscopy as well as X-ray diffractometry demonstrated that the chemical composition and microstructure of ZrO_2-Ni FGM distributed gradiently in stepwise way. The constituents, both zirconia and nickel, were continuous in microstructure everywhere, eliminating the macroscopic interface in traditional ceramics/metal joint. By means of finite element method, the thermal stress distribution in a layered ZrO_2/Ni material (non-FGM) and that in a ZrO_2-Ni FGM under an analogical condition with great temperature difference were preliminarily analyzed. It was shown that the thermal stress value in ZrO_2-Ni FGM remarkably decreased and distributed more smoothly in comparison with that in ZrO_2/Ni non-FGM. Obviously the ZrO_2-Ni FGM has the function of thermal stress relaxation.

Key words functionally graded material zirconia nickel microstructure thermal stress relaxation

1 INTRODUCTION

With the progress of aerospace technology, the property requirement for materials has been more and more severe. Under super-high temperature and great temperature gradient, it is quite necessary to develop a new type of heat-resisting material. A new material concept functionally graded material (FGM) has been proposed to meet the need[1, 2], which usually comprises different material constituents such as ceramics and metal. The ceramic side offers heat-resistance and the metal side provides mechanical strength and thermal conductivity. The continuous change of consititution between the two sides of FGM can eliminate the traditional joint interface and relax the thermal stress induced by the temperature difference in use. Various techniques of material preparation, such as physical and chemical vapor deposition, plasma spraying and powder metallurgy (P/M), etc. have been employed to the fabrication of FGM, in which P/M is one of the most basic routes for its unique advantage of control in composition and structure[3]. We have developed ZrO_2-Ni FGM by P/M method[4], and the aim of the present paper is to investigate its microstructural characteristics and preliminarly analyze the thermal stress relaxation behaviour by finite element method.

2 EXPERIMENTAL AND ANALYTICAL PROCEDURE

Partially stablized zirconia (PSZ, doped with 3% (in mole) Yttria) and nickel were chosen as the FGM components, whose physical properties were shown in the following Table 1. It may be seen that the thermal expansion coefficient and elastic modulus of PSZ are close to those of nickel, which is advantageous to mechanical and thermal matches between the two components.

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Table 1 Physical properties of the components in ZrO_2-Ni FGM

Material	Melting point / ℃	Density / $g \cdot cm^{-3}$	Thermal expansion coefficient / $K^{-1} \times 10^{-6}$	Thermal conductivity / $W \cdot (m \cdot K)^{-1}$	Elastic modulus / GPa	Poisson's ratio
PSZ	2 700	5.85	8.0~ 9.0	2.10	186.3	0.360
Ni	1 453	8.85	13~ 16	84.15	199.5	0.312

The raw materials are PSZ powder with average diameter of 0.5 μm and 99.8% in purity, and fogging nickel powder with 45 μm diameter and 99.5% purity. The raw powders with different PSZ/ Ni ratios were first blended by wet milling. After being dried, the mixed powders were filled sequentially with gradual changes in mixing ratio and compacted in a steel die. Then the green compact was hot pressed at 1 350 ℃, and the ZrO_2-Ni FGM plate (60 mm × 60 mm × 5 mm) was gained finally. Samples for microstructural inspection were cut with a diamond saw.

The microstructure and elemental distribution of FGM were observed by means of optical microscope and Hitachi S-570 scanning electron microscope (SEM). The phase composition and substructure of FGM were investigated by D/ max-rB X-ray diffractometer (XRD) and Philips CM-12 transmission electron microscopy (TEM), respectively. TEM samples were mechanically milled to 0.08 mm thickness and followed by ion thinning to transparency. The operating voltage of TEM was 120 kV.

The distributions of temperature and thermal stress in the FGM and a direct layered ZrO_2/ Ni material (non-FGM) under an analogically working condition were preliminarily analyzed by finite element method. The analogical environment was supposed that the ceramic side was heated to high temperature and the metal side was compulsorily cooled.

3 RESULTS AND DISCUSSION

3.1 Microstructure and compositional distribution

Fig. 1 (a) presents the optical macrophotograpy of the cross section in ZrO_2-Ni FGM. It is observed that the microstructure distributes with a stepwise gradient and gradually changes from nickel to PSZ ceramics. As shown in Fig. 2, the SEM line analysis of FGM reveals that the content of zirconium increases progressively from metal side to ceramic side, but that of nickel decreases relevantly. Clearly the chemical composition in the FGM exhibits graded distribution and meets the expected design in the main.

The microscopic observations demonstrate that the microstructure of each layer in sintered ZrO_2-Ni FGM is quite dense and homogeneous (Fig. 1(b)). However, the distribution form of the components in FGM varies with the compositional change as well. In Ni rich regions, nickel serves as matrix phase and displays typical network structure, in which the PSZ particles disperse and act as dispersion strengthening. In above 60% (in volume) PSZ regions, the continuity transition of the FGM components can be found. Nickel phase begins to change from connective distribution to dispersive one, but PSZ phase gradually changes from dispersive to connective conjugately. The dispersive distribution of Ni constituent in PSZ matrix can produce toughening effect of ductile particles. This microstructural transition agrees with the fractal analysis[5].

It is noteworthy that, even on the prestacked section where chemical composition stepwise jumped (marked "o" in Fig. 1), both ceramics and metal components are continuous in microstructure. Thus there is no macroscopic interface in FGM. This good continuity of microstructure can eliminate the disadvantage of traditional macroscopic interface in ceramics/ metal joint, and reflect the design idea of FGM.

3.2 Phase composition and substructure

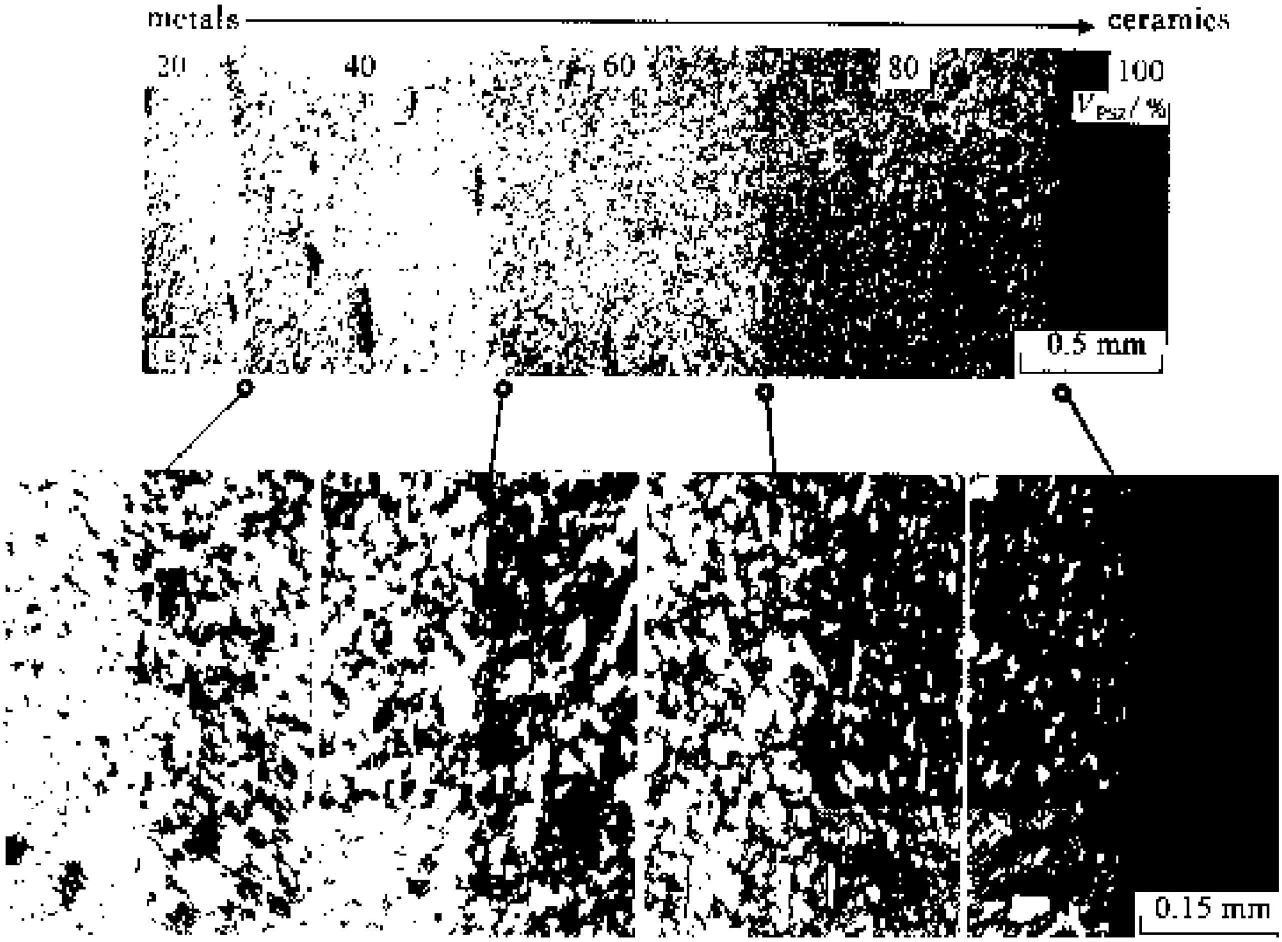

Fig. 1　Microstructure of the cross section in ZrO_2-Ni FGM

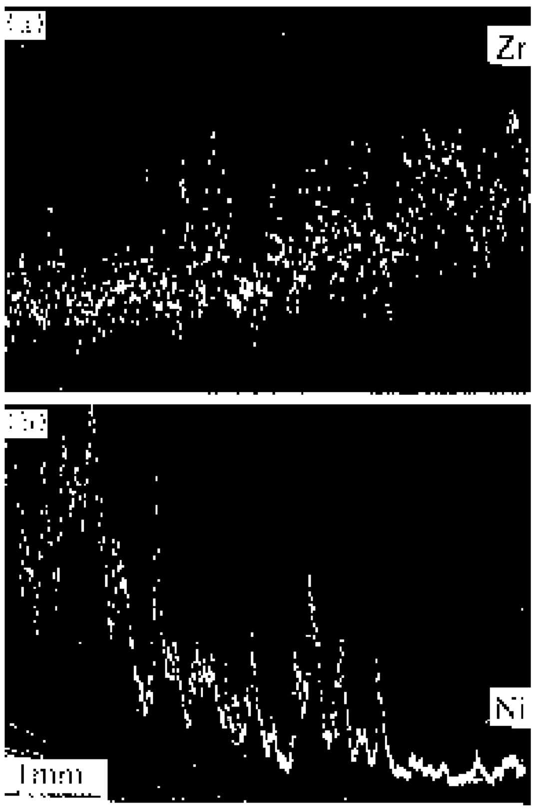

Fig. 2　SEM line analysis of ZrO_2-Ni FGM

As shown in Fig. 3, the X-ray diffraction pattern indicates that the PSZ component in ZrO_2-Ni FGM consists of the tetrogonal zirconia (t-ZrO_2) and a little monoclinic zirconia (m-ZrO_2), and Ni component exists in the form of element. It means that the ZrO_2 component is quite compatible with the nickel component in chemistry, and the FGM of ZrO_2-Ni system can be employed in high temperature environment which requires heat stabilization.

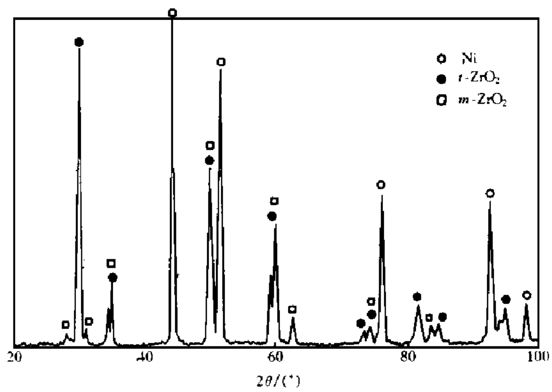

Fig. 3　X-ray diffraction pattern of ZrO_2-Ni FGM

The TEM observations, as presented in Fig. 4, further confirm the above XRD result. It can be seen that the t-ZrO_2 phase exists as fine grained structure (0.2 ~ 0.4 μm), and the grains are nearly equiaxed and rather uniform in size (Fig. 4(a)). The $t \rightarrow m$ martensite transformation has partially taken place in some large t phase grain in the cooling process after sintering. The substructure of m-ZrO_2 is twin (Fig. 4(b)), which is resulted from twinning of m-ZrO_2 to accommodate the shape change during $t \rightarrow m$ transformation. Owing to the volume expansion and the shear effect from t to m phase, further $t \rightarrow m$ transformation has been restrained by constraint of the mother phase matrix, and the majority of metastable t phase remains at room temperature. Those metastable t phase will transform to m phase induced by stress in use, which can absorb energy and act as transformation toughening[6, 7]. Another component nickel in FGM displays typical structure of annealing twin (Fig. 4(c)). It reveals that nickel had undergone obviously thermal-plastic flow during hot pressing, which promoted the densifying process of FGM, and then recrystallized and grown. Because of the lower energy of stacking fault, nickel is easy to form annealing twin, in which high density dislocations produced by shrinking as cooling can be found.

On the other hand, the mosaic structure of nickel and PSZ is formed due to the restraint of PSZ particles to the coarsing of nickel grains (Fig. 4(d)), which can obviously improve the strength and toughness of FGM. The PSZ/Ni phase interfaces are quite straight and seem to be directly connected (Fig. 4(d)) and no interface reaction is detected, which is in agreement with the XRD results.

Fig. 4 TEM photographs of ZrO_2-Ni FGM

(a) — t-ZrO_2; (b) — t + m dual phase mixture; (c) —Ni twin; (d) —Ni/t interface

3.3 Thermal stress analysis

The thermal stress in the plates of ZrO_2-Ni FGM and layered ZrO_2/Ni material (non-FGM) has been analyzed by finite element method, whose analysis model and temperature boundary condition are shown in Fig. 5. The analogical working environment is supposed that the ceramic side is exposed to high temperature to 1500 ℃, and the metal side is cooled to − 100 ℃. Assume that the other four edges of the plates are insulated and simply supported. The conditions as above can be simplified to the steady thermal conduction with one-dimension. Let it be supposed that the material is elastic under the above conditions and no plastic deformation occurs, and the material properties do not change with the temperature. The properties of composite mateial in graded layers are caculated by the mixture rules[8]. The FGM and non-FGM plates are divided into 840 and 640 meshes, respectively.

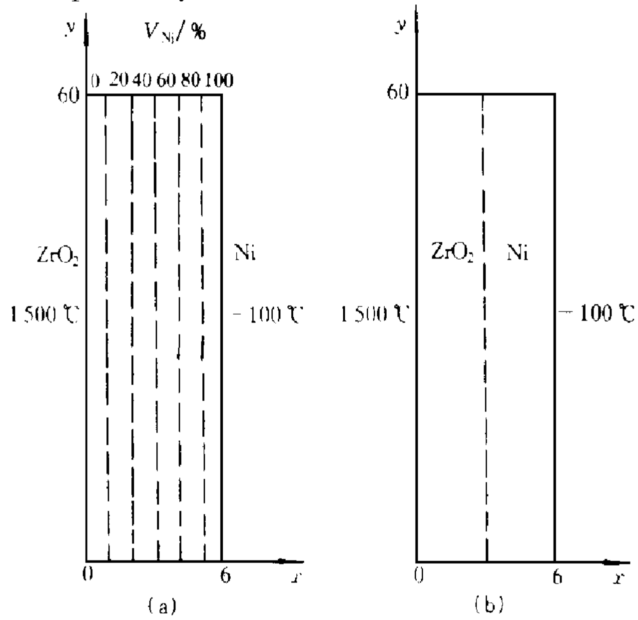

Fig. 5 Analysis model of thermal stress
(a) —FGM; (b) —non-FGM

Fig. 6 illustrates the caculated results of temperature and thermal stress fields under the analogical working condition. In FGM, the heated PSZ side is effectively cooled by conducting of the nickel constituent, due to the graded distribution of nickel component with good heat conductivity. As a result, the temperature varies more smoothly in FGM, decreasing the heat load. But the temperature distribution strikingly changes at ceramics/metal joint interface in non-FGM (Fig. 6(a)). It can be noted that the slope of temperature curve in FGM alters in some degree on the section between different gradient layers, which relates to the uncontinuous change of thermal conductivity induced by the stepwise graded distribution (refer to Fig. 1). At x = 2

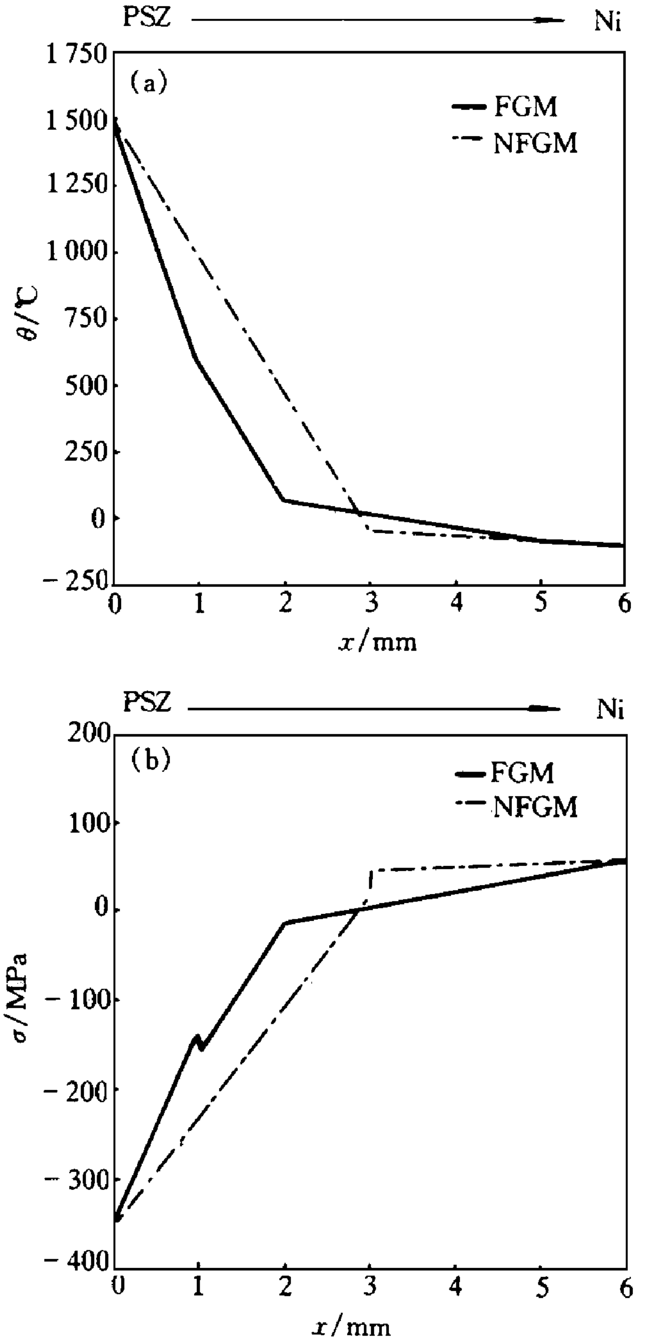

Fig. 6 Results of finite element method
(a) —temperature field;
(b) —thermal stress field

mm (i. e. the section between 80% PSZ layer and 60% PSZ layer), the slope change of temperature curve in FGM is more obvious, reflecting the continuity transition of FGM components. Based on the microanalysis (Fig. 1) the nickel component in 80% PSZ layer exists dispersively and its heat conductivity is mainly depended on the PSZ matrix with extremely low conductivity of heat. In 60% PSZ layer, the nickel begins to transit from dispersive phase to continuous matrix phase, and the heat conductivity greatly rises, which results in a sudden slope change of the temperature field.

As it is seen from Fig. 6(b), the varying tendency of stress field in FGM plate, which was simply supported at edges, is similar to that in non-FGM, transiting from compression state at ceramic side to tension state at metal side. That is decided by the two factors thermal expansion coefficient and temperature distribution. Though the thermal expansion coefficient of PSZ is smaller than that of nickel, the temperature of PSZ side is far higher than that of nickel side, so the thermal expansion of PSZ side is more than that of nickel side. As a consequence, the PSZ side is in compression due to the constraint of nickel side and the edges to be supported, but the metal side in tension is induced by the cooling shrinkage. This stress state will be of great advantage to develop the own properties of brittle ceramics and ductile metal constituents, which further reveals the resonableness of the match between PSZ and Ni components.

However, there are obviously different characteristics in thermal stress distribution of FGM and non-FGM plates (Fig. 6(b)). Corresponding to the temperature distribution, the PSZ/Ni joint interface in non-FGM bears quite high tensile stress, which will result in spallation at the ceramic/metal interface easily. Although the stress values of PSZ side and Ni side in FGM plate, under the same condition of steady thermal conduction and constraint at edges as above, are equal to those in non-FGM plate, the stress decreases remarkablely in FGM plate and is less than that in non-FGM plate everywhere (Fig. 6(b)). What is more important, graded consititution changes the distribution of thermal stress, which distributes more smoothly and eliminates failure possibility of material induced by the maxium interface stress. Obviously, the FGM has the function of thermal stress relaxation and the aim of FGM design is achieved.

4 CONCLUSIONS

(1) Hot pressed ZrO_2-Ni FGM exhibits graded distribution both in composition and microstructure, in which the components mixed uniformly and transited continuously. So the macroscopic interface of traditional ceramics/metal joint is eliminated.

(2) No reactant between ZrO_2 and Ni is detected in hot-pressed ZrO_2-Ni FGM, and the phase composition comprises nickel, tetragonal zirconia and a little monoclinic zirconia. The substructure of m-ZrO_2 is twin, and nickel also displays a typical structure of annealing twin.

(3) The analysis by finite element method demonstrates that the temperature varies more smoothly in FGM but strikingly changes at PSZ/Ni joint interface in non-FGM under the analogic working condition with great temperature difference. Accordingly, the layered PSZ/Ni plate bears quite high tensile stress on joint interface and will result in interface spallation. In contrast, the thermal stress in FGM plate decreases remarkablely and distributes more smoothly. Obviously the ZrO_2-Ni FGM has the function of thermal stress relaxation.

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