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Short communication

Effect of adding fiber on the microstructure and mechanical properties of $NiFe_2O_4$ composite ceramics

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Abstract

$NiFe_2O_4$ composite ceramics reinforced with carbon fibers (C_f), nickel-coated carbon fibers (NiC_f) and ZrO_2 fibers (ZrO_{2f}) were fabricated by the conventional high-temperature solid state reaction at 1300 °C for 6 hs. The effects of the three kinds of fibers on microstructure and mechanical properties of $NiFe_2O_4$ composite ceramics were investigated. The fracture surfaces of $NiFe_2O_4$ composite ceramics were observed by scanning electron microscopy (SEM). Elemental analysis of micro-structural phases was performed using energy dispersive spectroscopy (EDS), attached with SEM. Flexural strength by three-point bending techniques and fracture toughness were measured. It was found that leaving holes after reactions between the carbon fiber and matrix led to decreased mechanical properties. The ZrO_{2f}-doped composite had an improved fracture toughness of 3.05 ± 0.1 MPa $m^{1/2}$ compared to $NiFe_2O_4$ composite ceramics having 1.91 ± 0.2 MPa $m^{1/2}$ due to fiber debonding, fiber pull-out and fiber bridging as well as microcracking toughening. The results presented here show that ZrO_{2f} can serve as a potential toughening agent for $NiFe_2O_4$ composite ceramics.

Keywords: A. Calcination; B. Fibers; C. Mechanical properties; E. Electrodes

1. Introduction

Since the Hall–Héroult process was brought into researchers′ view in the 1960s, various researches have been carried out to find a novel material serving as inert anode to produce environment-friendly O_2 gas during aluminum electrolysis instead of greenhouse gases (CO_2), fluorocarbons (CF_4, C_2F_6) and sulfurous gases (SO_2, CS_2, H_2S). Up to now, we have not found appropriate electrode materials to meet commercial requirements. Recently, most researchers have concentrated on $NiFe_2O_4$ composite ceramics, because they have the following properties: good resistance against fluoride melts, high resistance against oxygen coming from the anode, adequate strength at high temperatures and high thermal stability [1–4]. Nonetheless, we all know that their low fracture toughness is still the obstacle preventing ceramic matrix composites from being widely used. Their poor toughness and inability to withstand heat shocks make the anode crack, and the purity of the electrolytic Al is influenced negatively when this kind of anode material was used in aluminum electrolysis. The tests with inert anode were carried out in 6-KA pilot cells with the support of the US Department of Energy. They announced that the experiment on inert anode was delayed due to its cracking [5]. Therefore, how to improve the toughness of $NiFe_2O_4$ composite ceramics has become a challenging task for us. Now the main methods are to introduce a toughening phase into the ceramic matrix, such as particles, fibers and whiskers [6–12]. Various studies have been carried out to improve the mechanical properties of $NiFe_2O_4$ composite ceramics. Zhang et al. [13,14] reported that the mechanical properties of $NiFe_2O_4$ were improved due to the addition of the SiC_f, but they also found that the $NiFe_2O_4$ matrix easily reacted with SiC_f above 1180 °C. Ma et al. [15] reported that the bending strength of samples is improved by about 22% with 3 wt% copper-coated carbon fiber compared to that of $NiFe_2O_4$ composite ceramics. However, the fracture toughness of the copper-coated carbon fiber-doped composite was not evaluated. Hua et al. [16,17] investigated the best preparation

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technique of synthesis of $ZrO_{2f}/NiFe_2O_4$ composite ceramics, and also studied the corrosion behavior of ZrO_{2f}–$NiFe_2O_4$ inert anode in cryolite molten salt. The results show that the electrolytic corrosion rate of anode samples containing 3 wt% ZrO_{2f} is 2.2 mg/(cm^2 h), which is much lower than its static thermal corrosion rate. For all that, reports on $NiFe_2O_4$ composite ceramics toughened by fibers are very limited.

In this work, powder metallurgy methods were adopted to prepare $NiFe_2O_4$ composite ceramics. In order to improve the fracture toughness of $NiFe_2O_4$ ceramics, we tried to introduce toughening phases, such as fibers, including C_f, nickel-coated carbon fibers and ZrO_{2f}, into the ceramic matrix to toughen the $NiFe_2O_4$ spinel based ceramics. The purpose of this work is to investigate the effect of three different kinds of fibers on microstructure and mechanical properties of the $NiFe_2O_4$ spinel based ceramics, respectively.

2. Experimental procedure

2.1. Synthesis

Powders of Fe_2O_3 (0.5–1.0 μm, purity > 99.3 wt%), NiO (0.5–1.0 μm, purity > 99.98 wt%, excessive 17 wt%) were used as major raw materials to synthesize $NiFe_2O_4$ matrix material. The mixture of the two powders was ground in distilled water by ball-milling for 24 h, and then the mixture was dried thoroughly. The dried mixture was calcined at 1200 °C in air for 6 h to produce the $NiFe_2O_4$ spinel matrix material. The calcined ceramic matrix was crushed and screened, and then the powders with particle size under 100 meshes were milled with carbon fibers, nickel-coated carbon fibers and ZrO_{2f} in distilled water for 6 h, respectively. The slurries with different components were dried thoroughly and were ground with 4 vol% polyvinyl alcohol (PVA) binder and pressed into blocks (60 mm × 15 mm × 8 mm) at 200 MPa. The green samples with carbon fibers, nickel-coated carbon fibers and ZrO_{2f} were sintered in air at 1300 °C for 6 h, respectively, and then furnace-cooled to ambient temperature.

2.2. Characterization

Specimens were polished and ultrasonically cleaned. The microstructure of fracture sections of samples after three-point bending tests was observed by scanning electron microscopy (SEM, SSX-550, Japan) with simultaneous chemical analysis by energy dispersive spectroscopy. The phase composition was determined by X-ray diffraction (XRD, Rigaku, Dmax-rb). The bulk density of the samples was determined using the Archimedes technique with water as the immersing medium. Flexural strength in a three-point configuration was tested using an electron omnipotence machine NSTRON-4206 on a 60 mm × 15 mm × 8 mm chambered bar with a span of 30 mm and crosshead speed of 0.05 mm/min. Peak load was obtained when the sample was fractured and the average value of six samples was obtained. Fracture toughness (K_{IC}) was evaluated with a span of 30 mm and a crosshead speed of 0.05 mm/min using a single-edge notched beam test on 60 mm × 15 mm × 8 mm test bars on the same jig used for the flexural strength.

3. Results and discussion

3.1. Microstructure and phase composition

3.1.1. Effect of carbon fibers on $NiFe_2O_4$ composite ceramics

As we know, carbon fibers have been extensively used to reinforce ceramic–matrix composites because of their high thermal shock resistance, good bonding strength and modulus, high chemical stability and toughness. SEM micrographs of fracture sections of $NiFe_2O_4$ spinel based ceramics and $C_f/NiFe_2O_4$ composite ceramics are presented in Fig. 1. The grain sizes of the ferrite particles sintered in air at 1300 °C were in the range 2–5 μm, and the grain sizes of C_f-doped samples sintered in air at 1300 °C were in the range 5–7 μm. The SEM micrographs of fracture sections reveal that incorporation of carbon fibers is beneficial to enhance grain growth of $NiFe_2O_4$. In addition, no trace of the carbon fibers can be seen in the ceramic composites, but it is interesting to note that

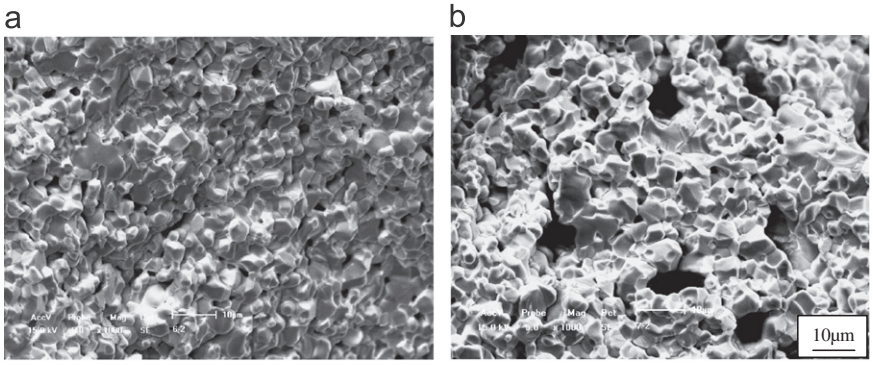

Fig. 1. SEM micrographs of fracture sections of samples: (a) $NiFe_2O_4$ spinel based ceramics and (b) $C_f/NiFe_2O_4$ composite ceramics.

significant irregular pits and the leaf-like surface were left in the sintered samples (see Fig. 1(b)). The XRD plot, obtained from the polished surface of the sample is presented in Fig. 2. The major crystalline phases are found to be $Ni_{0.6}Fe_{2.4}O_4$, Fe_2O_3, NiO and Fe_3O_4. In addition, a new phase Fe_3N is also detected. These phenomena may be attributed to the reactions between C (the main component of carbon fibers) and NiO and Fe_2O_3 [18–20] at 1300 °C as listed in Table 1. From Table 1, the thermodynamic calculation indicates that the Gibbs free energy changes of the two reactions are all negative values at 600–1600 K, illustrating the existence of the two reactions. Reactants (Ni and Fe) will continue to be oxidized generating the corresponding oxide. The results that are obtained show that carbon fibers cannot be stable in $NiFe_2O_4$ composite ceramics after sintering at 1300 °C due to their reactions with the main phases in matrix material.

3.1.2. Effect of nickel-coated carbon fibers on $NiFe_2O_4$ composite ceramics

Nickel and copper are the two main metals used as coating on carbon fibers. Urena et al. [21] found that copper coatings easily dissolve above 950 °C, so we choose chemical nickel-plating on carbon fibers in order to protect carbon fibers from being oxidized. Fig. 3 shows the SEM micrograph of a fracture section of nickel-coated carbon fibers/$NiFe_2O_4$ spinel based ceramics and related EDS analysis. Significant irregular pits and a long groove of remaining fibrous textures were observed. In addition, it is very interesting to observe that adding nickel-coated carbon fibers can bring abnormal grain growth of $NiFe_2O_4$. Some grain sizes of nickel-coated carbon fiber-doped samples changed from equixed structure (~3 μm) to rod-shaped (~14 μm in length and ~5 μm in width), as seen in Fig. 3(b). The reason for this is that sintering process is liquid phase sintering and not solid phase sintering due to melting of metal nickel. Although the melting of nickel is at 1453 °C higher than the sintering temperature (1200 °C), we gain alloy coatings (low melting point) during the chemical nickel-plating process [22,23]. The melting metal nickel after the disappearance of carbon fibers will react with Fe_2O_3 and O_2; the reactions will be

$$3Ni(s) + Fe_2O_3(s) = 3NiO(s) + 2Fe(s)$$

$$2Ni(s) + O_2(g) = 2NiO(s)$$

The XRD spectrum obtained from the polished surface of the composite ceramic is shown in Fig. 2. The XRD diffraction peak of the $NiC_f/NiFe_2O_4$ is almost the same as that of the $C_f/NiFe_2O_4$. When a combination of XRD and SEM analyses was used, the results showed that electroless nickel plating on carbon fibers did not protect carbon fibers from being oxidized and can bring abnormal grain growth instead. It can be attributed to two main reasons: (1) the differences in thermal expansion coefficient between nickel and carbon fibers. Carbon fibers have a high thermal anisotropy, for example, the thermal expansion coefficients are $(0.67–1.0) \times 10^{-6}\ °C^{-1}$ and $(8–27) \times 10^{-6}\ °C^{-1}$ in the axial and radial directions, respectively [24,25], which are far below that of nickel ($13 \times 10^{-6}\ °C^{-1}$). Generation of thermal stress, which is originated from the difference in thermal expansion coefficients between nickel-coated and carbon fiber during cooling from the high temperature of 1300 °C to room temperature, destroyed the good adhesion between fibers and metal coating. Exposed carbon fibers will react and then disappear. (2) Nickel-coating being on the carbon fiber surface layer has been damaged by an external force before sintering, such as stirring to make the fibers disperse homogenously in the composite.

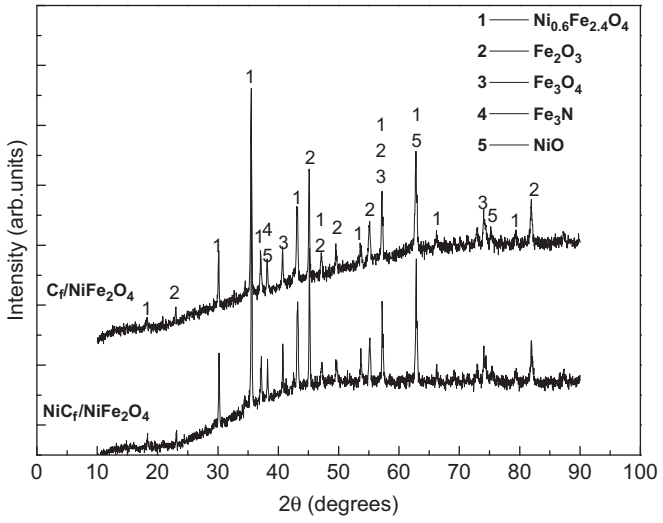

Fig. 2. XRD spectra obtained from the polished surfaces of the $C_f/NiFe_2O_4$ and $NiC_f/NiFe_2O_4$ composite ceramics.

3.1.3. Effect of ZrO_{2f} on $NiFe_2O_4$ composite ceramics

It is well known that ZrO_{2f} retains good thermal stability (2677 °C, melting point) and strong resistance to oxidation at a higher temperature. Hua et al. [16] reported that ZrO_{2f} can be stable in $NiFe_2O_4$ spinel based ceramics when

Table 1
Reactions between C and NiO, Fe_2O_3 and the Gibbs free energies at 600–1600 K.

Reaction equations	ΔG_T (kJ mol^{-1})					
	600 K	800 K	1000 K	1200 K	1400 K	1600 K
$C + 2NiO = 2Ni + CO_2$	−25.35	−60.79	−95.54	−129.74	−163.50	−196.92
$3C + 2Fe_2O_3 = 4Fe + 3CO_2$	140.03	36.41	−63.54	−163.12	−262.24	−360.16

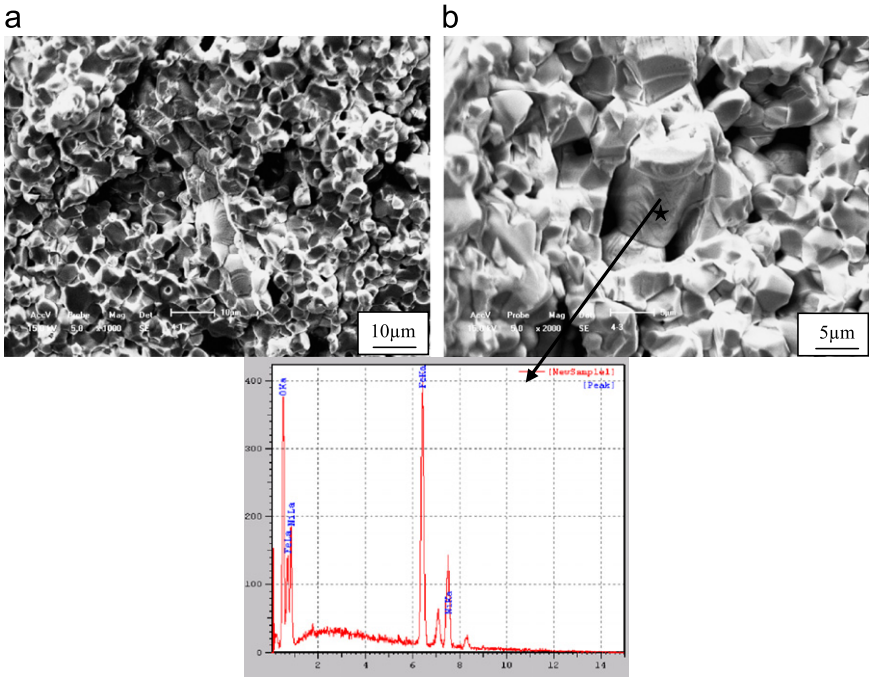

Fig. 3. SEM photographs and EDS analysis of $NiC_f/NiFe_2O_4$.

Table 2
Density and mechanical properties of samples.

Materials	Density ($g\,cm^{-3}$)	Relative density (%)	Flexural strength (MPa)	Fracture toughness ($MPa\,m^{1/2}$)
3 wt% C_f	4.22	83.6	40 ± 2	1.71 ± 0.2
3 wt% NiC_f	4.50	84.0	36 ± 3	1.65 ± 0.09
$NiFe_2O_4$	4.54	85.1	53 ± 1	1.91 ± 0.05
1 wt% ZrO_{2f}	4.36	83.2	57 ± 2	2.23 ± 0.06
2 wt% ZrO_{2f}	4.23	81.8	61 ± 3	2.57 ± 0.04
3 wt% ZrO_{2f}	4.17	80.9	67 ± 1	3.05 ± 0.1
4 wt% ZrO_{2f}	3.84	76.5	55 ± 3	2.60 ± 0.09

sintering temperature is 1300 °C. Moreover, there are no chemical reactions between ZrO_{2f} and $NiFe_2O_4$. The variation of the flexural strength and fracture toughness of the prepared $ZrO_{2f}/NiFe_2O_4$ samples with ZrO_{2f} content is shown in Table 2. It is clear that the flexural strength and fracture toughness for sample doped with ZrO_{2f} are apparently higher than those of the undoped sample. This implies that addition of ZrO_{2f} has an effect on mechanical properties of $ZrO_{2f}/NiFe_2O_4$ composites. The flexural strength and fracture toughness of the composite increase first and then decrease with an increase of ZrO_{2f} content. Maximum flexural strength of 67 ± 1 MPa and fracture toughness of 3.05 ± 0.1 MPa $m^{1/2}$ are obtained by addition of ZrO_{2f} up to 3 wt%. Addition of ZrO_{2f} content beyond 3.0 wt% shows a decrease of flexural strength and fracture toughness Miyazaki et al. [26] reported that the improvement in fracture toughness of the composite ceramics was originated mainly from the "stress-induced" transformation of zirconia phase. SEM photographs of fracture sections and EDS analysis of samples are presented in Fig. 4. ZrO_{2f} roots can be clearly seen in the fracture surface of the $ZrO_{2f}/NiFe_2O_4$ spinel based ceramics, and the average particle size of ZrO_{2f}-doped samples was ~2 μm, their grain sizes were smaller than those of undoped ceramic samples. It suggested that the addition of ZrO_{2f} resulted in decreased sizes of the grains [19]. The microstructure of the composite ceramics is thus refined, the strength is consequently enhanced. When the content of the ZrO_{2f} is beyond 3.0 wt%, the mechanical properties decreased greatly. It is mainly attributed to low density of the composites (see Table 2). Fig. 5 indicates that no other phases are detected except for $NiFe_2O_4$, ZrO_2 and NiO. The combined XRD and SEM analysis shows that ZrO_{2f} can reinforce $NiFe_2O_4$ spinel based ceramics after sintering at 1300 °C. Moreover, the best mechanical properties of the composite ceramics were obtained with 3 wt% ZrO_{2f} additive.

3.2. Mechanical properties

Results of the density and mechanical properties are listed in Table 2. From the result, it is clear that incorporation of fiber increased the difficulty of densification in ceramic matrix. The value of the mechanical properties of C_f-doped and NiC_f-doped samples was lower than that of undoped ceramic samples. In particular, the minimum value of flexural strength of 36 ± 3 MPa and

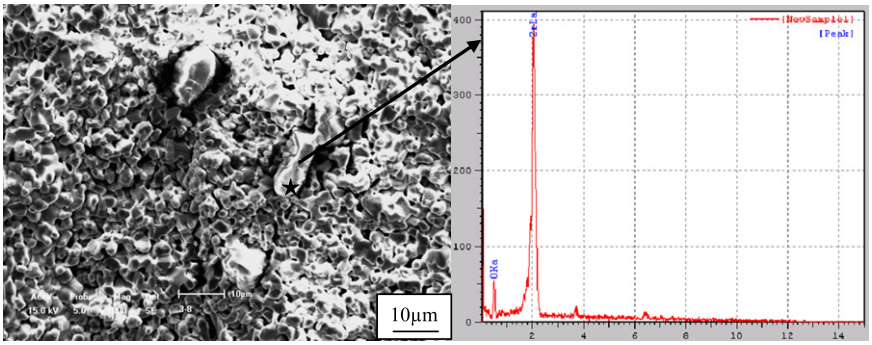

Fig. 4. SEM images of $ZrO_{2f}/NiFe_2O_4$ composite ceramics.

fracture toughness of 1.65 ± 0.09 MPa $m^{1/2}$ were gained for $NiC_f/NiFe_2O_4$. That is because abnormal grain growth and non-uniform distribution of grain size can directly result in decrease of mechanical properties [27]. Generally speaking, the flexural strength of ceramic materials mainly depends on several factors, such as ceramic porosity, grain size and microstructure [28,29]. Eqs. (3) and (4) illustrate that increasing grain size and porosity can weaken material strength:

$$\delta = \delta_0 + Kd^{-1/2} \quad (3)$$

$$\tau = \tau_0 \exp(-bp) \quad (4)$$

where δ is the material strength, δ_0 is the yield stress, K is a constant and d is the grain size, p is the porosity, τ is the strength of the sample with porosity of p, τ_0 is strength for sample without pores, and b is a constant. Eqs. (3) and (4) have further proved that the mechanical properties of ceramic samples containing carbon fibers or nickel-coated carbon fibers were lower than those of undoped samples. The flexural strength of the $ZrO_{2f}/NiFe_2O_4$ spinel based ceramics was 67 ± 1 MPa, which was statistically $\sim$26.4% higher than that of samples without fibers. Compared with the carbon fibers and nickel-coated carbon fibers, the increase in the flexural strength of the $ZrO_{2f}/NiFe_2O_4$ spinel based ceramics was ascribed to effective toughening mechanisms of the ZrO_{2f}. Improvement in the fracture toughness from 1.91 ± 0.05 MPa $m^{1/2}$ for $NiFe_2O_4$ spinel based ceramics to 3.05 ± 0.1 MPa $m^{1/2}$ for $ZrO_{2f}/NiFe_2O_4$ spinel based ceramics was also the result of the addition of ZrO_2 fibers. In order to further investigate the effect of ZrO_2 fibers on toughening mechanisms, representative microstructures of $ZrO_{2f}/NiFe_2O_4$ are given in Fig. 6, and the fiber bridging and holes left after ZrO_2 fiber pull-out can be seen. ZrO_{2f} in the composite materials first debonded in the fiber-matrix when it was subjected to external load. Then slipping occurred between the fibers and matrix, and finally ZrO_{2f} fibers were pulled out near the crack tip. It is believed that it is effective for the external load to transfer to the fibers for ZrO_{2f}-doped samples in the matrix, thus promoting successful toughening. At the same time, microcracking can be clearly seen

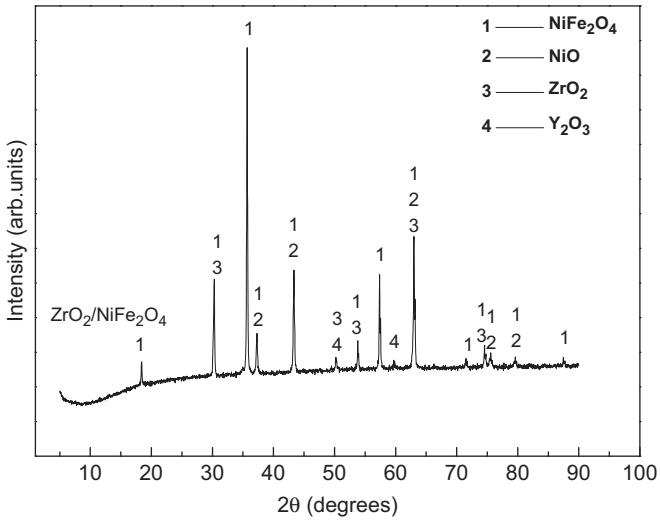

Fig. 5. XRD spectra obtained from the polished surfaces of the $ZrO_{2f}/NiFe_2O_4$ composite ceramics.

from Fig. 6(b). Meguid [30]and Basua et al. [31] reported that microcracking can develop near the tip of a main crack as a result of the combined effect of externally applied loads and localized residual stresses. It is believed that they can deplete the energy during crack propagation. So this near-tip stress induced microcracking has been regarded as another toughening mechanism.

4. Conclusions

Fibers, including carbon fibers, nickel-coated carbon fibers and ZrO_{2f}, reinforced $NiFe_2O_4$ spinel based ceramics have been fabricated by the high-temperature solid state reaction at 1300 °C for 6 h. The results show that carbon fibers and nickel-coated carbon fibers cannot effectively reinforce $NiFe_2O_4$ spinel based ceramics, and ZrO_{2f} can serve as a potential toughening agent for $NiFe_2O_4$ composite ceramics. Measurements of mechanical properties indicate that flexural strength and fracture toughness of the composite ceramics with 3 wt% ZrO_{2f} achieve maximum values of 67 ± 1 MPa and 3.05 ± 0.1 MPa $m^{1/2}$,

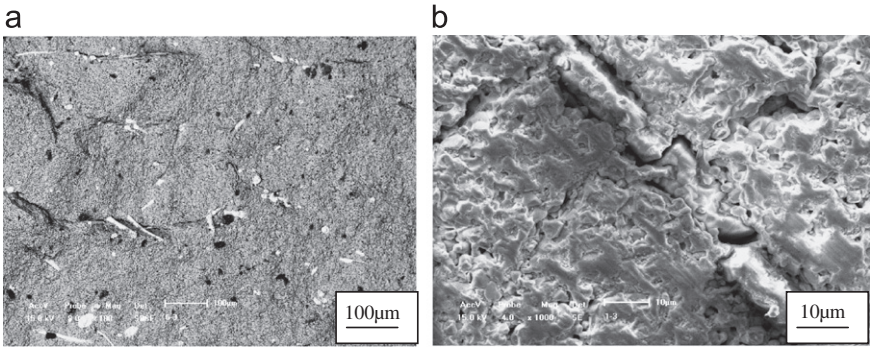

Fig. 6. SEM images of $ZrO_{2f}/NiFe_2O_4$ composite ceramics: (a) the fractured surface $ZrO_{2f}/NiFe_2O_4$ and (b) microcracking in $ZrO_{2f}/NiFe_2O_4$ composites.

respectively. The increase in toughness was attributed mainly to the addition of the ZrO_{2f}, its main toughening mechanisms were microcracking toughening, fiber pull-out and fiber bridging.

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