Article

Fabrication of ZrO_2/Ni functionally graded materials by underwater-shock compaction

Akira Chiba*, and Yasuhiro Morizono*

ZrO_2/Ni Functionally Graded Materials (FGMs) were fabricated by newly developed underwater-shock compaction technique. The experimental assembly consists of three parts viz., an explosive container, a water tank and a powder container. The energy of shock wave increases by the convergence of shock wave due to the reduction of the cross section area of water tank. The increased shock pressure and longer duration of the pressure pulses facilitate the compaction of difficult-to-compact powders to near theoretical density. The pressure obtained was estimated to be 4 GPa. The 6- to 11-layered FGMs were fabricated. The 11-layered FGMs were subjected to cyclic differential thermal loading test and were found to withstand the thermal stress successfully.

1. Introduction

Recent advances in technology require structural materials that can be used in severe environments. As super thermal protective materials for future aerospace transportation system, some type of functionally graded materials have been proposed due to endure the sudden thermal shock loading or to get good high temperature strength [1].

Functional Graded Materials are characterized as materials having continuously varying material property from one surface to the other and are prepared through several methods such as CVD, PVD, ion plating, plasma spraying, sintering and self-propagating high temperatures synthesis [2]. The gradient changes of composition can reduce or eliminate specific interfaces between constituent materials such as ceramics and metals.

Shock compaction is a well known technique to produce bulk materials from powders without detriment to the basic properties of the participant powders [3,4]. However, it was hitherto difficult to obtain sound specimens without any cracks and/or central hole by using an axisymmetric explosive compaction technique.

The authors have shock compacted powders such as ZrO_2 and Si_3N_4 ceramic powders, utilizing underwater-shock wave generated by the detonation of explosives and by using the water as a pressure medium. They have reported that sound specimens can be fabricated without using any sintering additives. The advantage of shock compaction technique is that the compact with full density can be fabricated within microseconds.

The purpose of the present study is to establish a methodology to fabricate ZrO_2/Ni FGMs by using the underwater-shock compaction technique mentioned above and to investigate the microstructures and thermo-mechanical properties of ZrO_2/Ni FGMs.

2. Experimental procedure

2.1 Materials

Partially stabilized zirconia powder (TZ-3Y) was supplied from Tosoh Co.,Tokyo Japan. The content of Y_2O_3 in the powder was 3.64 (mass%) and the average particle size was about 0.3μm. Ni powder was supplied from Nilaco Co., Ltd., Tokyo, Japan and the average particle size was about 5μm.

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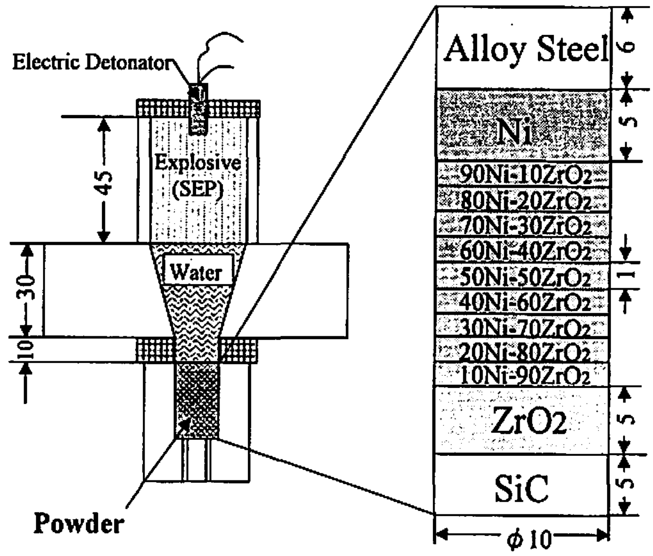
*Kumamoto University, Kumamoto 860-855, JAPAN
Telephone number and FAX : 096-342-3718
E-mail: chiba@gpo.kumamoto-u.ac.jp



[Mixing ratio :vol.%, Unit :mm]

Fig. 1 Schematic illustration of underwater-shock compaction assembly and stacking situation for 11-layered ZrO_2/Ni FGMs.

2.2 Underwater-shock compaction assembly

The assembly used in present study consists of an explosive container, a water tank and a powder container as illustrated in Fig. 1. When 11-layered ZrO_2/Ni FGMs were produced, the mixed powders with the ZrO_2: Ni ratios, viz., 9:1, 8:2, 7:3, · · 7: 3, 2:8, 1:9, were loosely packed in the powder container (Figure 1). Hard steel powder on the top was used to prevent the scattering of FGM powders from the powder container and SUS304 powder in the bottom was used for momentum trap. All containers were made of mild steel. The intensity of the shock wave of the explosive detonation increases by the convergence of shock wave due to the reduction of cross section area and the reflection of the shock waves on the conical wall of the water tank. Varying the mass and the detonation velocity of the explosive and the conical angle of the water container can easily control the magnitude of shock pressure and the shock duration.

The explosive used in the present study was SEP (provided by Asahi Kasei Co., LTD.) and consisted mainly of nitric ester and its detonation velocity was 6900 m/s. The shock compacted ZrO_2 monolithic material and ZrO_2/Ni FGM was sintered at various temperatures

2.3 Estimation of shock pressure

The shock velocity at the powder container was measured by the ion gap method and the shock pressure was calculated using the equation by Cole [5] and Penny and Dasgupta [6] expressing the relationship between the shock pressure and

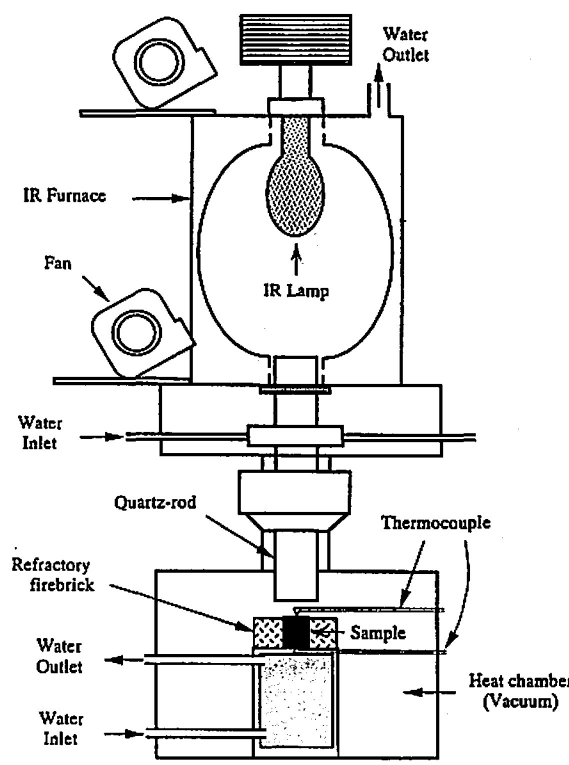


Fig. 2 Assembly for differential thermal loading test.

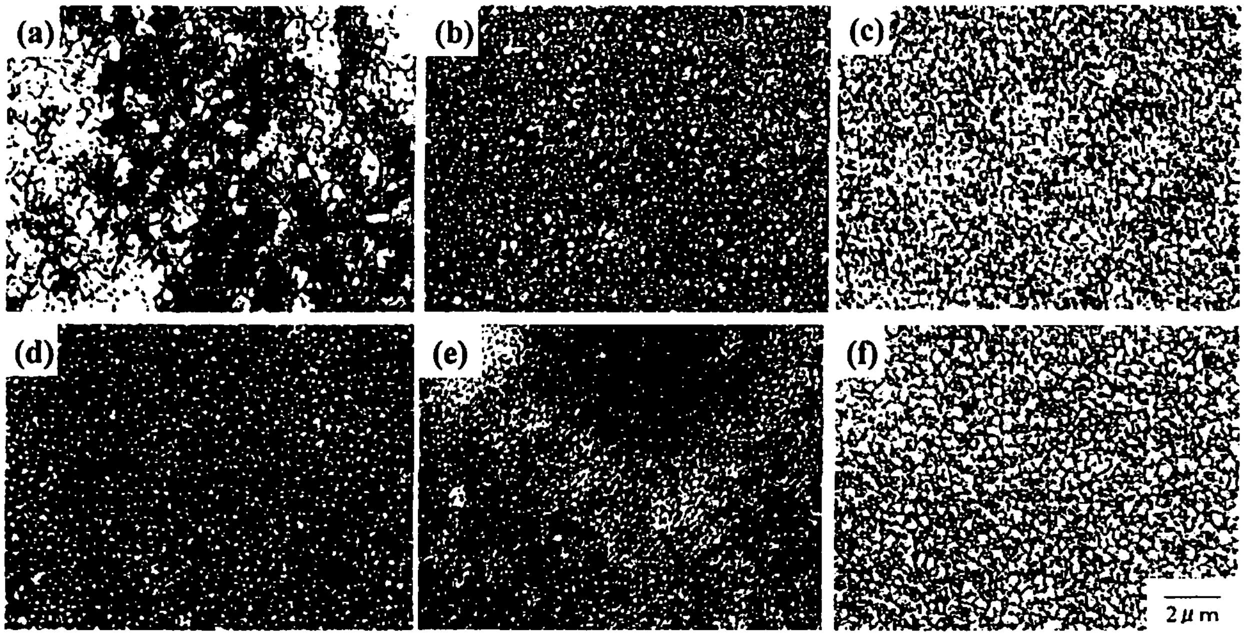


Fig. 3 SEM micrographs of as-compacted and post-sintered ZrO_2 monolithic materials, (a) as-compacted state, and post-sintered states, (b) at 1473K for 4h, (c) at1573K for 2h, (d) at 1573K for 4h, (e) at 1673K for 2h, and (f) at 1673K for 4h, respectively.

velocity in water. The shock velocity was estimated by the arrival time difference of shock wave between pins of different heights.

2.4 Microstructural investigation and thermomechanical test

Microstructural characterization of the compacted ZrO_2 monolithic materials and ZrO_2/Ni FGMs was performed by optical microscopy, scanning electron microscopy with EDX analysis. The mechanical properties of ZrO_2/Ni FGMs were investigated by fracture toughness. Fracture toughness of ZrO_2 monolithic compacts was measured by the indentation fracture (IF) method. Thermomechanical property of the FGMs was investigated by the cyclic differential thermal loading test (Fig. 2).

3. Experimental results and discussion

3.1 Microstructure and mechanical properties of ZrO_2 monolithic compact

Figure 3 shows the microstructures of the as-compacted and post-sintered compacts sintered at various sintering temperatures. Many voids are observed in the as-compacted specimen. Good bonding between powders was not achieved as-compacted state. Post-sintering is, thus, inevitable to get sound compacts. Figure 3 shows that sintered samples showed higher density with increasing sintering temperatures. After sintering at 1673K for 2h, the sample shows good bonding between powder particles and very few voids. But ZrO_2 compact showed grain growth after the sintering at 1673K for 4h.

Figure 4 shows the relationship between relative densities and sintering conditions of ZrO_2 monolithic compacts and Fig 5 reveals the relationship between hardness and sintering conditions of ZrO_2 monolithic compacts. In the as-

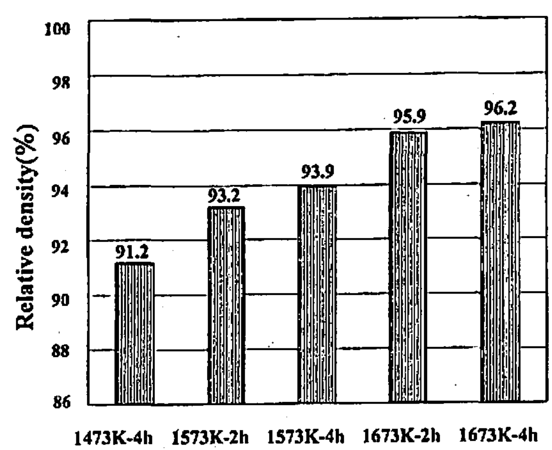


Fig. 4 Relationship between relative density and sintering conditions in ZrO_2 monolithic material.

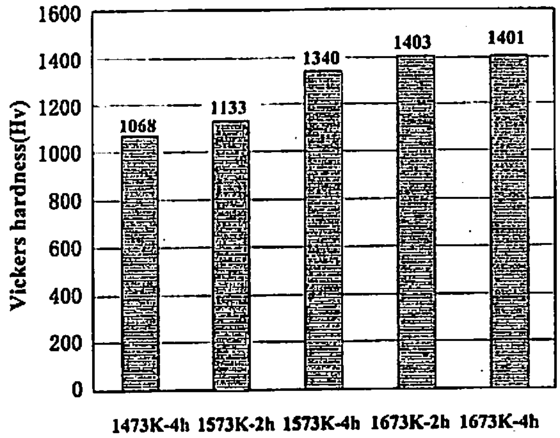


Fig. 5 Relationship between hardness and sintering conditions in ZrO_2 monolithic material.

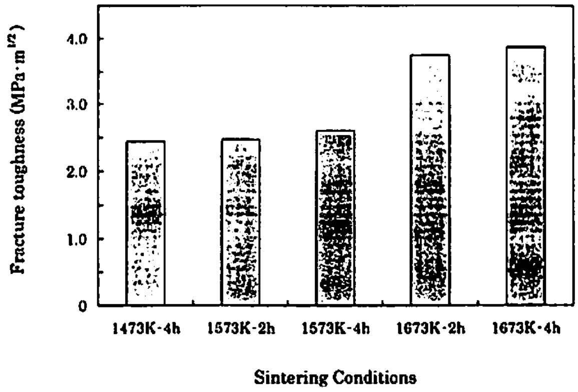


Fig. 6 Relationship between fracture toughness and sintering conditions in ZrO_2 monolithic material.

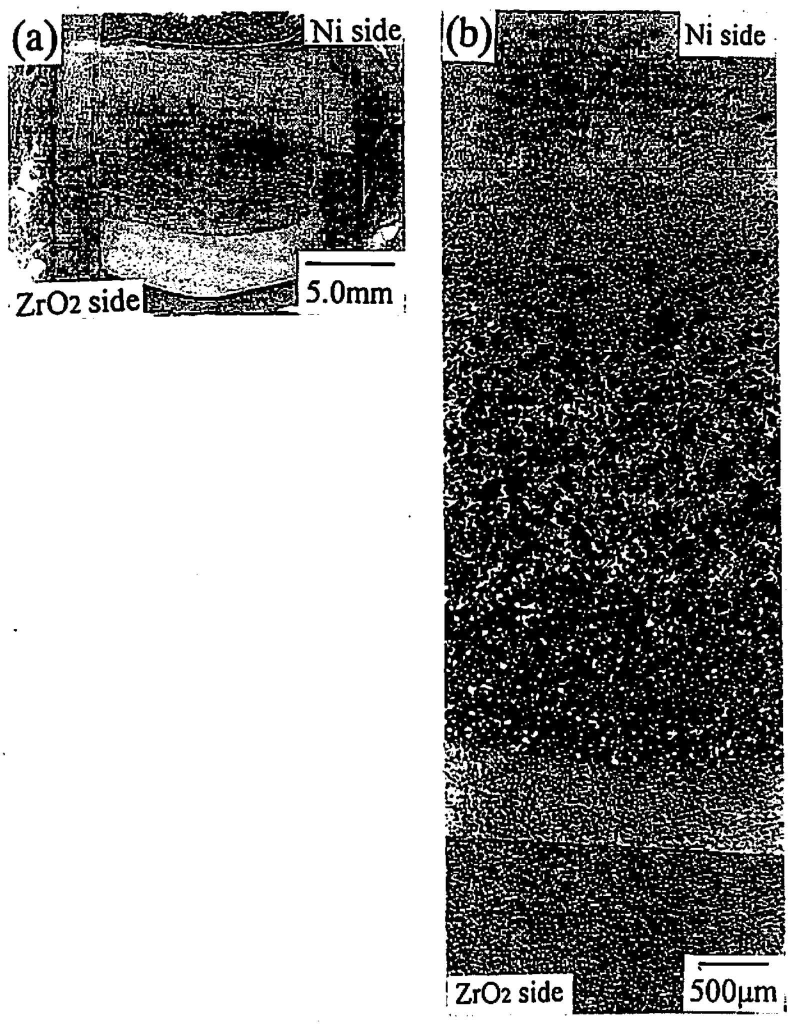


Fig. 7 External view and optical micrograph of cross section of ZrO_2/Ni FGM cut parallel to the shock compaction direction.

compacted state, the relative density was about 65%. After sintering it is evident that relative densities increase with increasing sintering temperatures and with increasing sintering times and the relative density after sintering at 1673K showed about 96%. The hardness variations also

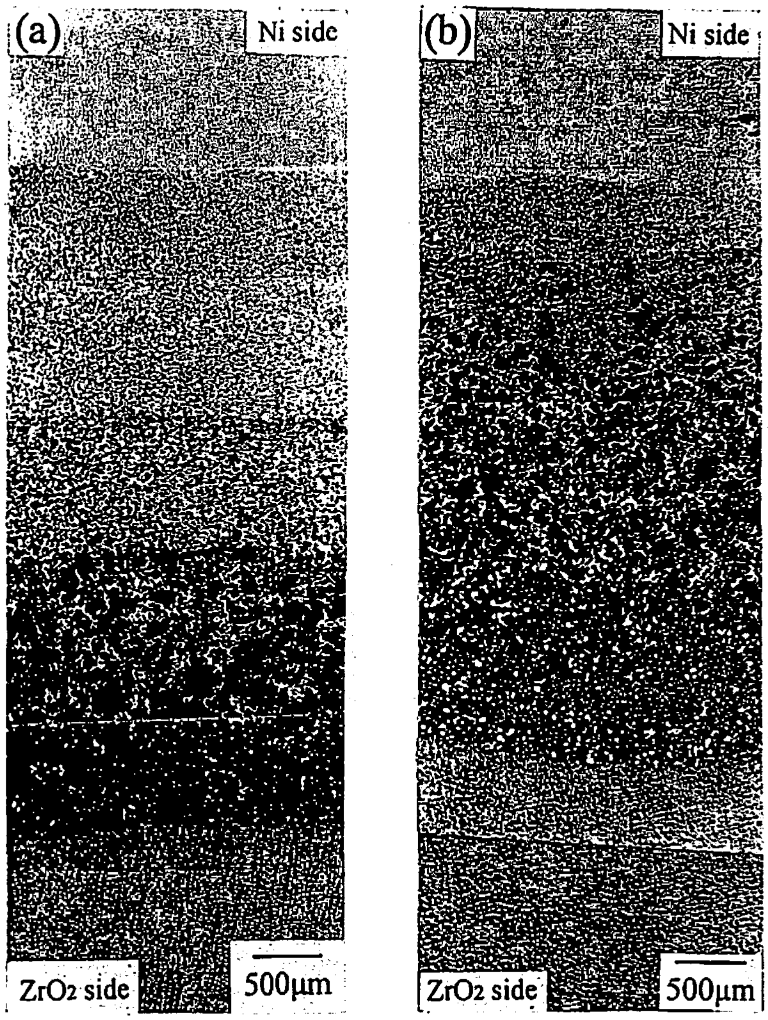


Fig. 8 Optical micrographs of ZrO_2/Ni FGM compact in as-compacted state, (a) 6-layered FGM, and (b) 11-layered FGM.

showed the same tendency as relative density. The hardness of the specimen after sintering at 1673K is about 1400 Hv, which is almost the same as the hardness of the commercial ZrO_2 materials (1300Hv).

Figure 6 shows the relationship between the fracture toughness and sintering conditions. The fracture toughness of the compacts sintered at 1673K is almost same value as the ZrO_2 materials commercially available.

The optimum sintering condition to get sound specimens in the present experiment is found to be 1673K and 2h.

3.2 Microstructure and mechanical properties of ZrO_2/Ni FGMs

Figure 7 shows the external view of the cross sections of ZrO2/Ni FGM sectioned parallel to the shock compaction direction. The thickness of the compact is reduced about one half of the loosely packed state.

Figure 8 shows the optical microstructures of 6 layered and 11-layered FGM compacts in the as-compacted states respectively. Upper part indicates Ni side and the bottom part is ZrO_2 side. Both compacts were found to be free of cracks and tears. They are found to have undergone continuous compositional changes.

Figure 9 shows the optical microstructures of both compacts after the sintering at 1673K for 2h. Some cracks and tears are observed in the specimen of 6-layered compact but in the specimen of 11-layered compact micro cracks and tears are not seen.

Thermal stress formed between the layers during sintering treatment is more than the

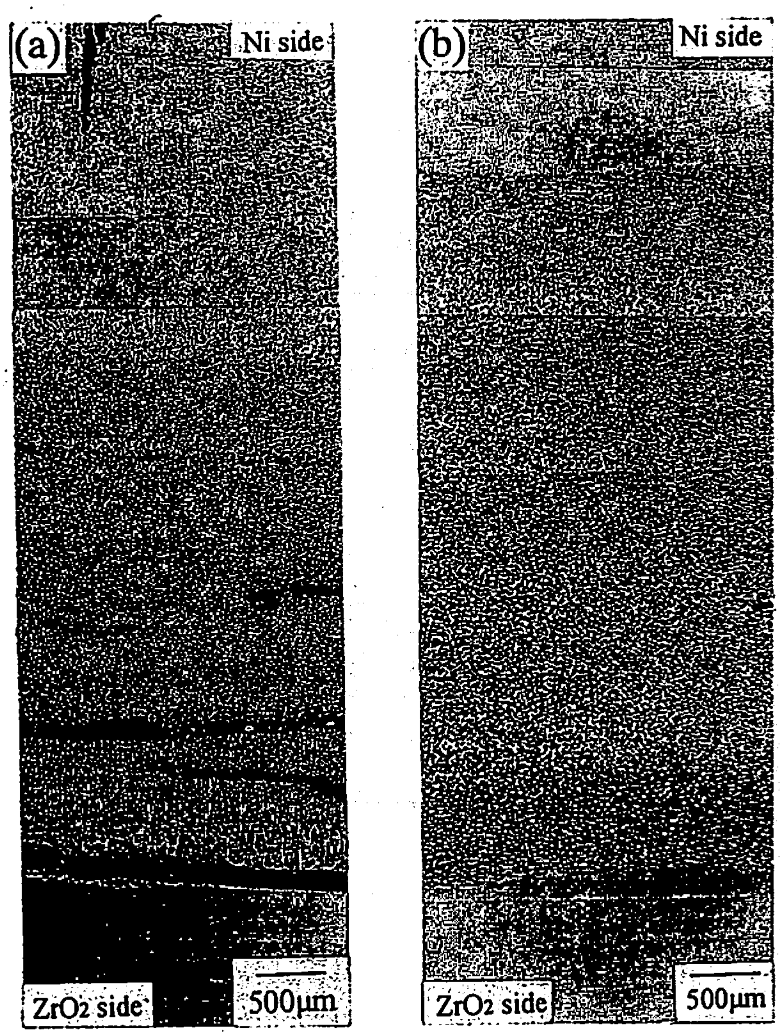


Fig. 9 Optical micrographs ZrO_2/Ni FGM compacts sintered at 1673K for 2h, (a) 6-layered FGM and, (b) 11-layered FGM.

bonding strength in 6-layered compact, and is less than the bonding strength in 11-layered compact.

Figure 10 shows the result of EDX analysis of ZrO_2/Ni FGM. Figure 10(a) shows the secondary electron image. Figure 10(b) and (c) show the characteristic X-ray images of Zr and Ni respectively. Continuous concentration change is observed.

Figure 11 shows an X-ray diffraction pattern of the compact whose mixing ratio (ZrO_2: Ni) is 1:1 and was sintered at 1673K for 2h. Peaks of Ni and ZrO_2, which exists as tetragonal crystal, are observed. Traces of any reaction products between Ni and ZrO_2 are not seen. This implies that sintering does not produce any reaction between Ni and ZrO_2 in the compacts.

Figure 12 shows the relation between hardness and Ni content in the as-compacted and post-sintered (1673 K at 2 h) FGMs. The hardness of as-compacted Ni is observed to be higher than that of the post-sintered compacts and this is due to effect of work hardening introduced by shock compaction. Further, sintering reduces the hardness by the release of the hardening stress. The hardness values of the samples post-sintered are directly correlated to the ZrO_2 contents and showed higher values than as-compacted states. The relative density after the post-sintering shows more than 95% relative densities (Fig.13).

The cyclic differential thermal loading test was performed by using an infrared ray furnace. In one cycle, the surface of ZrO_2 side was heated to 1273K for 2min. and cooled in air. On the other hand, the surface of Ni side was cooled with water flow and maintained at 573K, therefore the temperature difference was 700K. After 30 thermal cycles, there were no cracks and tears in 11-layered ZrO_2/Ni FGMs (Fig. 14). This result indicates that

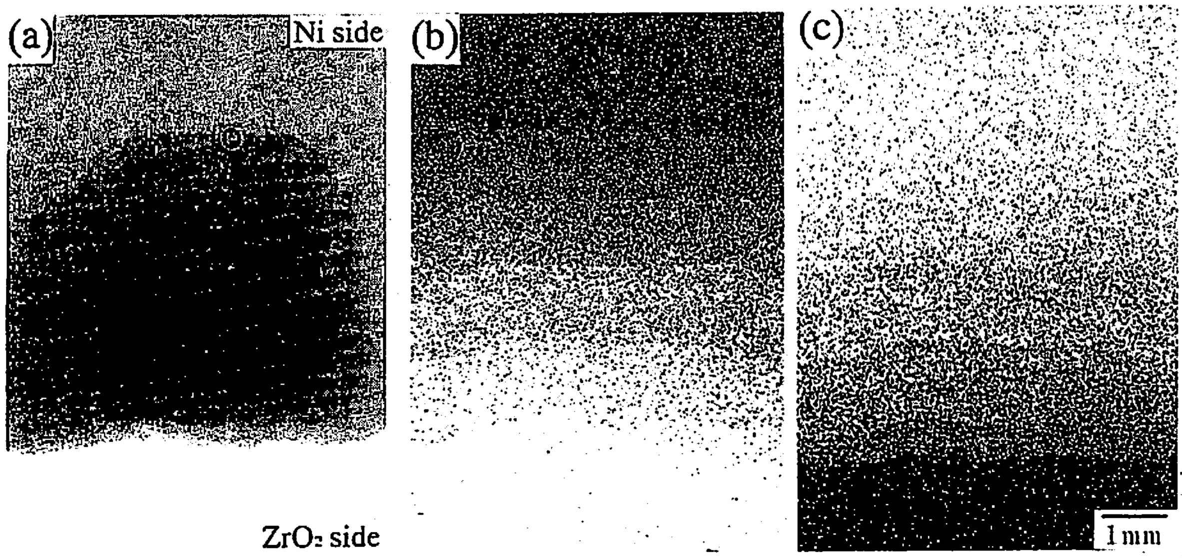


Fig. 10 EDX analysis of ZrO_2/Ni FGM compact after sintering at 1673K for 2h, (a) secondary electron image, (b) and (c) characteristic X-ray images of Zr and Ni respectively.

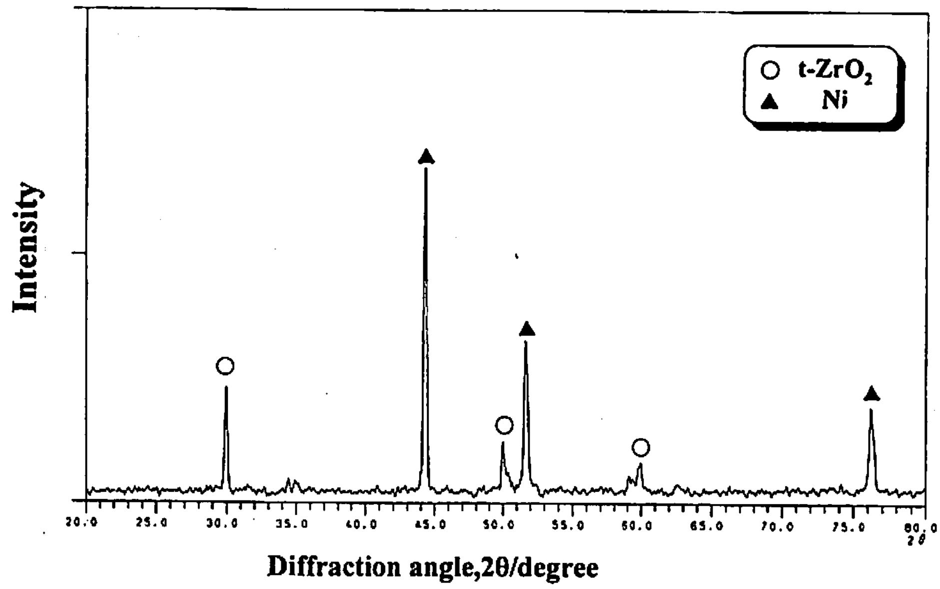


Fig. 11 X-ray diffraction pattern of ZrO_2/Ni compact (ZrO_2: Ni = 1:1) sintered at 1673K for 2h.

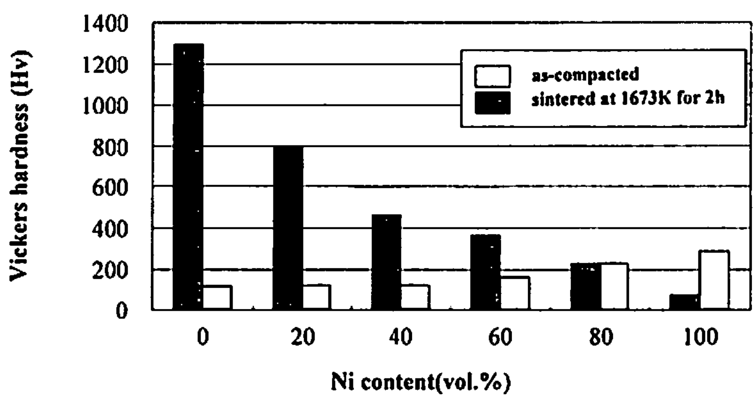


Fig. 12 Relationship between hardness and Ni contents in ZrO_2/Ni FGM compacts in as-compacted state and in sintered state at 1673K for 2h.

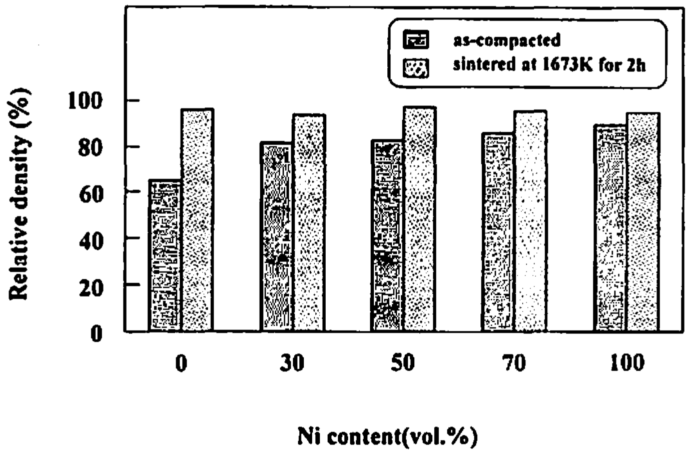


Fig. 13 Relationship between relative density and Ni contents in ZrO_2/Ni FGM compacts in as-compacted state and in sintered state at 1673K for 2h.

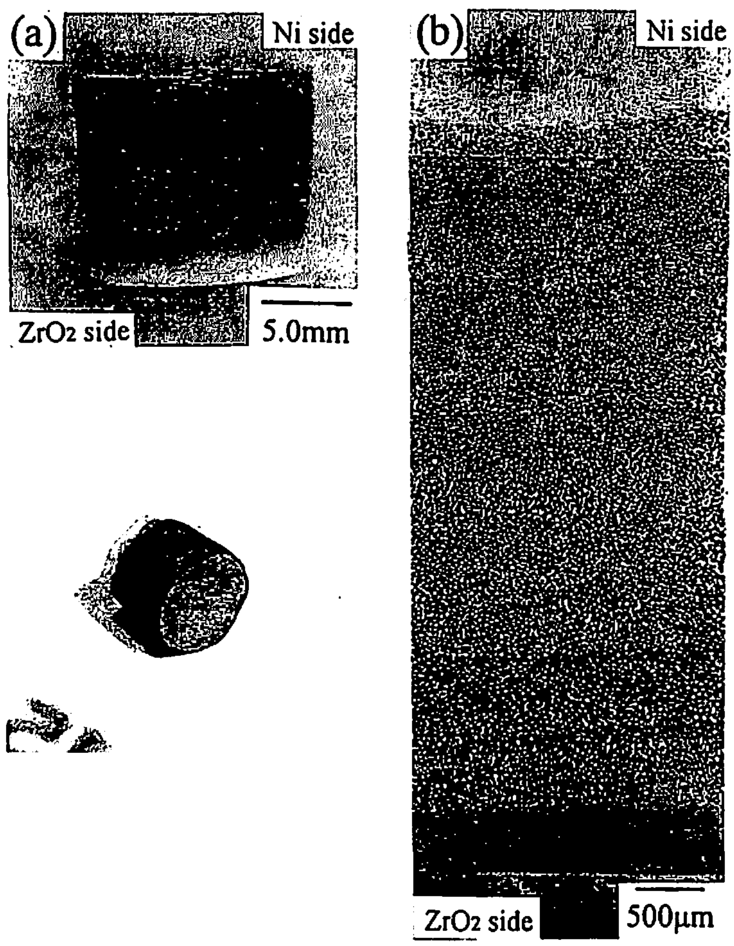


Fig. 14 (a) External view and (b) optical micrograph of the cross section of sintered ZrO_2/Ni FGM after differential thermal loading test (30cycles).

the residual thermal stress between monolithic ZrO_2 and Ni was fairly reduced by the presence of the composition gradient layers.

4. Conclusions

ZrO_2/Ni FGM was fabricated successfully by underwater-shock waves generated by the detonation of explosives. Water was used as the pressure-transmitting medium.

(1) ZrO_2/Ni FGM was fabricated without any binding additives. No cracks or tears were observed in the specimens. The specimen showed continuous compositional variation after heat treatment.

(2) ZrO2/Ni FGM was post-sintered at 1673K for 2h. Sintering treatment does not produce any reaction between Ni and ZrO_2 in the compacts.

(3) The cyclic differential thermal loading test was performed. The temperature difference between ZrO_2 side and Ni side was 700K. After 30 thermal cycles, there was no cracks and tears in the 11-layered FGM. The residual thermal stress in the ZrO_2/Ni FGMs was reduced by the presence of the composition gradient layers.

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