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Experimental study on mechanical properties of Ni/TiO_2 FGM processed by pressureless sintering technique

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Abstract. In the present work, an experimental investigation and characterization of Functionally Graded Materials (FGMs) is carried out. The Nickel (Ni) and Titanium dioxide (TiO_2) materials are taken as principal materials and the powder metallurgy technique is used for the preparation of specimens. The Nickel powder has a particle size of 20 μm with more than 99% purity and Titanium dioxide powder having 35 μm particle size with more than 99% purity is used for this study. The FGM specimens are prepared with five layers, with pure Nickel on one side and pure Titanium dioxide on opposite side of the specimen; and the three intermediate layers comprising of mixture of Nickel and Titanium dioxide. The five layered FGM samples are prepared with one inch diameter round die and the compacted samples are heated up to 1200°C in an inert gas (argon) atmosphere. The microscopic result shows that the microstructure of Ni/TiO_2 FGM has varied layer-by-layer and the interface between the layers are observed. As a part of mechanical characterization, both green and sintered FGM sample densities are measured for each layer of the sample. The Rockwell hardness test method is used to find the hardness of the each layer in the samples. Linear shrinkage of the specimen is calculated with the help of green sample dimensions. The Compression test is conducted on the sintered specimens by using universal testing machine; stress-strain behaviour and maximum stress reported.

Keywords. Functionally Graded Materials (FGMs); nickel; titanium dioxide; pressureless sintering; characterization.

1. Introduction

Functionally Graded Materials (FGMs) are non-homogeneous and advanced composites that consist of more than single component characterized by variation in material properties from one end to the other end [1]. The changes in properties are due to gradual variation of volume fraction of constituent materials from one surface to other. Metal–ceramic FGMs are expected to remove or reduce the residual stresses which cause cracking in the materials for the period of cooling from high temperatures and improve the bonding strength between the metal and ceramic materials [2]. The FGMs are made by varying the microstructure from one surface to another surface with a specific gradient. This is the reason for FGMs to have many advantages over the laminated composites [3].

One of the advantages of FGMs is that they can survive in environments with high temperature gradients, while maintaining structural integrity [4]. FGMs can be made a best promising material for human tissue implantation, due to its excellent biocompatibility, harmlessness, and corrosion resistance [5]. FGMs have the ability to inhibit crack propagation, due to this property FGMs are useful in defense applications and as a penetration resistant material it can be used for armor plates and bullet-proof vests [6].

FGMs can act as thermal barrier and can be used in energy conversion devices. They can also be used as protective coating on turbine blades in gas turbine engine [7, 8]. FGMs have many areas of applications like cutting tool insert coatings, nuclear reactor components, automobile engine components, tribology, turbine blade, heat exchanger, sensors, etc. [9–11].

Literature on FGM manufacturing process is reviewed. An apparatus for depositing the graded powder mixtures of two components has been presented by Olivier Gillia *et al* [12]. Direct laser metal sintering (DLMS) procedure is a revolutionary technique to build dental implants by Traini *et al* [13]. A novel processing procedure (homemade fabrication system) for the fabrication of graded metal/intermetallic materials is shown by Durejko Tomasz *et al* [14]. A number of processing techniques are available for producing FGMs and the powder metallurgy process is one of the approachable methods for fabricating components with gradual properties by gradually mixing two or more types of powders. The composition, microstructure and shape

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forming of the specimens are easily controlled by using the powder metallurgy technique.

The prime objective of this work is to fabricate bulk layered FGMs and to study the properties, in this scenario powder metallurgy FGM manufacturing technique is used to create the specimens keeping in view of above mentioned benefits. The FGM specimens (Ni/TiO_2) are fabricated with the help of pressureless sintering technique, as it produces residual stress-free samples under controlled porosity and it is easily available and more economical. Ni/TiO_2 is a novel structural/functional material that combines the merits of both ceramics and metals. The melting points of both Ni and TiO_2 are close and 1200°C is considered as optimal sintering temperature in the study.

2. Specimen preparation

Powder metallurgy is one of the best processing methods for the preparation of FGM specimens and the Ni powder (above 99% purity, average size: 20 μm), TiO_2 powder (above 99% purity, average size: 35 μm) are used as the raw materials. The composite layers are distributed based on the percentage of each material presented by volume. Weights of the powders are measured with the help of volume of the die and the densities of each powder. After weighing the powders for specific composite layer, ball mill is used for uniform blending of the powders for 24 hours and paraffin wax is used as a binder for the mixtures.

After blending process, the mixed powders are dried in an oven at 150°C for one hour to remove the moisture and to produce dry powders. The blended powders are compacted in High Strength Steel (HSS) die with a diameter of one inch for the cold pressing. The FGM samples are prepared by stacking the powders in layer-by-layer with volume change from pure TiO_2 side to pure Ni side through three consecutive internal layers with 30%, 50%, and 70% of Ni in the volume. Hydraulic press is used to compact the specimen with a load of 20 tons. Each layer is pre compacted before stacking the next layer, to maintain uniform compositional distribution. The arrangement of graded layers is shown in figure 1.

The compacted samples are placed in a graphite plate and Pressureless sintering technique is used to sinter the green specimens. The measured temperature variations in sintering process are shown in figure 2. The specimens are heated from room temperature to 1200°C in an electric furnace with a heating rate of 5°C. The heating process is carried out in four steps and the holding time for each step is 15 min. The holding time plays a vital role in sintering to produce specimen grains with approximately even shape and high density with less porosity in the sample. The furnace temperature reaches to 1200°C at 285 minute, at this stage the specimens are maintained at same temperature for 90 min., after that the heaters of the furnace are turned off and cooling process is started. The total sintering

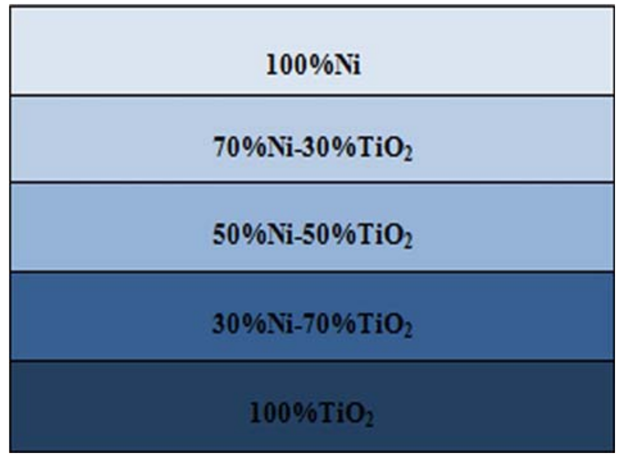

Figure 1. The arrangement of graded layers in 5 layered Ni/TiO_2.

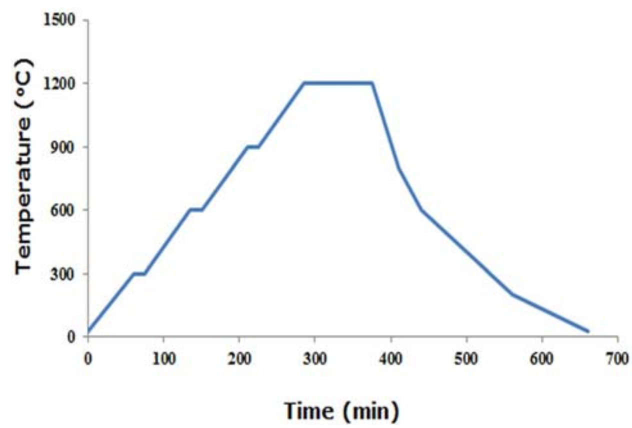

Figure 2. Measured temperature variations in sintering process.

process is carried out in an argon atmosphere maintaining one bar pressure throughout the sintering process.

3. Microstructure and mechanical characterization

The micro structure of the five layered FGMs is studied with Scanning Electronic Microscope (SEM). The compositional gradient along the thickness direction is also observed by using SEM images. Energy Dispersive Spectrometry (EDS) technique is used to determine the composition of the integral materials in FGM. The relative mechanical properties of Ni/TiO_2 composites are measured to define the mechanical property distributions along the thickness of five layered FGM. The green and sintered densities of the samples are measured respectively by using the dimensional methods.

The Rockwell hardness test is conducted as per ASTM E-18 standards, to know the hardness of each layer of sintered specimen. Linear shrinkage of each layer in sintered FGM is also measured. Compression Strength is measured by using universal testing machine and the specimen is made as per ASTM D-695. The average of three trails for all tests is calculated for five layered FGM and the results are shown in table 1.

4. Results and discussion

The five layered Ni/TiO_2 FGM specimens are prepared by using pressureless sintering technique. The microstructure, density, hardness, shrinkage and compression strength are measured for the sintered specimens and the results are discussed in detail in the following sections.

4.1 *Microstructure distribution*

The Ni/TiO_2 FGM specimens are cut into uniform sections and the surface of each section is sand down and polished with emery paper to visualize the microstructure. The microstructure of five layered FGM specimen is observed by using SEM images and is shown in figure 3. The EDS analyses of each phase in sintered layers and cross section of FGM is shown in figure 4. The composition of nickel and titanium dioxide varies from one end to the other and the weight percentages of elementary materials are achieved approximately in all layers. The average grain size of each sintered layer is measured using SEM analysis and shown in table 1. The quantitative measurement of compositional variation along the thickness direction is mentioned in table 2. The microstructural characterization shows that the distribution of ceramic reinforcement in each FGM layer is uniform. The interface of the compacted layers is observed and the metallic material is changed from matrix phase to fiber and the ceramic material is changed from fiber to matrix phase in the FGM. The micrograph shows that the five layers of the FGM metal to ceramic. From the results, it is observed that the composition varies from pure metal phase to ceramic phase and both phases are distributed evenly in the intermediate layers. The bonding of metal and ceramic phases is good in third layer, i.e., 50% metal and 50% ceramic layer compared to other layers and some thermal crackings are observed in ceramic rich regions. The thermal cracking in ceramic rich regions is attributed to the rate of cooling during the specimen preparation process.

4.2 *Density*

In fabrication of FGM, the powders are blended and compacted with hydraulic press due to this powder particles are held together by cold welds, it gives green strength to the specimen. In sintering, components undergo solid state sintering in multiple phases, during the heating nickel particles start wetting and come together due to capillary force. The grain boundary diffusion and highest densification rate at lowest grain growth is occurred over the temperature range of 900°C to 1200°C. The green and sintered density of the specimen is measured by dimensional method. The diameter of each layer is measured with outside callipers and thickness is measured with scale. The density is measured with the help of weight to volume ration. The green and sintered density of the specimens is plotted for each layer in Ni/TiO_2 system and is shown in figure 5. Almost full densification can be achieved for the pure materials compared with intermediate layers after sintering. The percentage of theoretical density for both green and sintered samples is shown in table 1. Thickness of each layer before and after sintering is measured and shown in table 3. The compact thickness of the specimen before sintering is 8.3 mm and after sintering is 6.3 mm. The density is increased with metal composition and high density is noticed at pure metallic layer after sintering. Above 90% of theoretical density is observed after sintering at the pure metallic layer.

4.3 *Hardness*

The Rockwell hardness at different layers of the sintered specimen is measured and the results shown in figure 6 as a function of TiO_2 present in Ni/TiO_2 system. The ball indenter of 1/16 inch size is used for this test and the applied load is 100 kg with 30 s of holding time to evaluate the hardness of sintered specimen. The layer-wise hardness values of the sintered specimen are shown in table 1. The

Table 1. Layer-wise properties of 5 layered Ni/TiO_2 FGM.

Layers	Average Grain Size (μm)	Density (% TD)		Hardness (HRB)	Shrinkage $(d_1 - d_0)/d_0$
		Green density	Sintered density		
Layer-1	21	66.46	91.18	68	0.176
Layer-2	23	64.17	86.68	72	0.133
Layer-3	24	61.93	83.77	80	0.09
Layer-4	26	58. 61	76.46	89	0.043
Layer-5	30	54.80	70.52	96	0.015

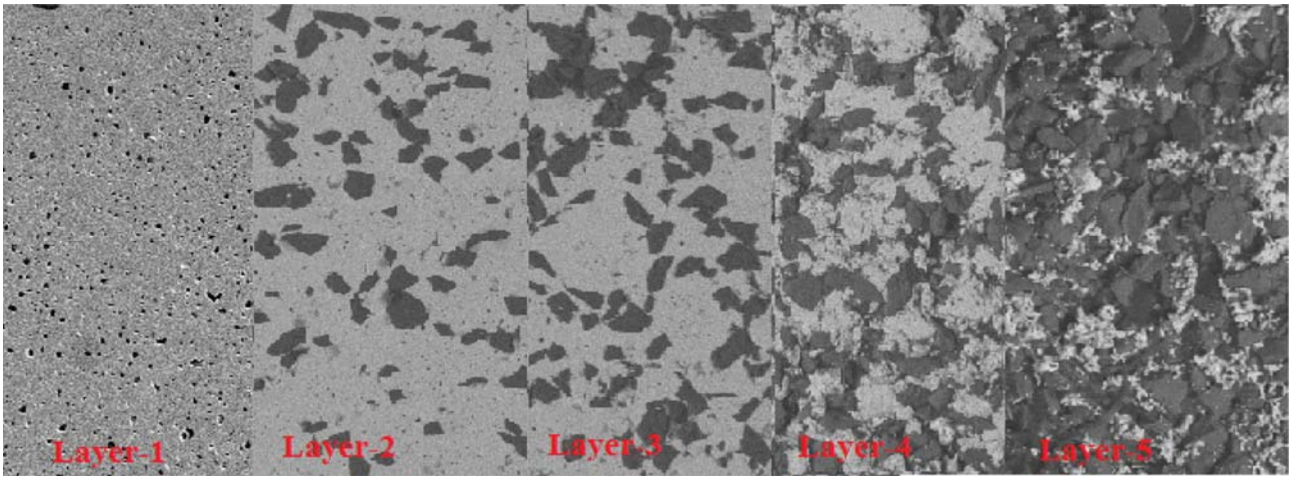

Figure 3. The microscopic images of 5 layered Ni/TiO_2 FGM.

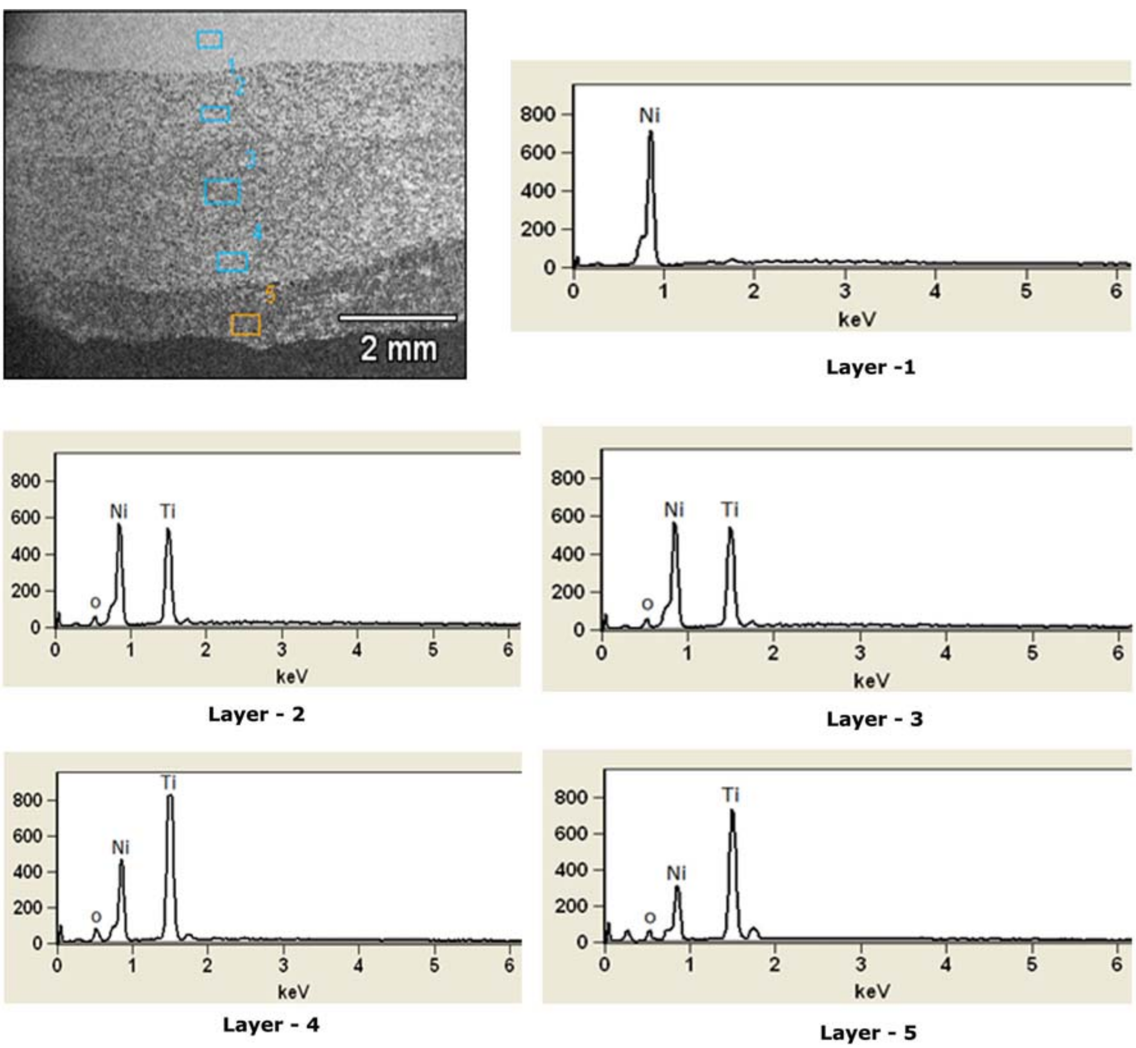

Figure 4. Microstructure of the polished cross-section of the Ni/TiO_2 FGM with EDS analyses.

Table 2. Quantitative measurement of compositional variation.

Layer	Composition
Layer-1	99.5% Ni
Layer-2	71.4% Ni–28.5 TiO_2
Layer-3	50.3% Ni–49.4% TiO_2
Layer-4	29.1% Ni–70.8% TiO_2
Layer-5	3.5% Ni–96.2% TiO_2

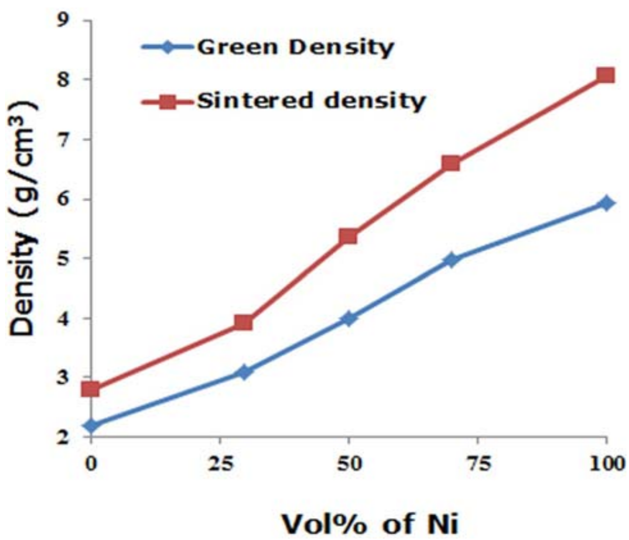

Figure 5. Density of 5 layered Ni/TiO_2 FGM.

Table 3. Layer thickness before and after sintering.

Layer Thickness	Before Sintering (mm)	After Sintering (mm)
Layer-1	1.5	1.1
Layer-2	1.6	1.2
Layer-3	1.6	1.3
Layer-4	1.7	1.3
Layer-5	1.8	1.4

results show that the hardness of each layer in the specimen is varying from 68 to 96 HRB and The hardness of the layers increases with increase of TiO_2 content in the layers.

4.4 *Shrinkage*

Shrinkage plays an important role in evaluating the quality of the specimens made by pressureless sintering technique. Figure 7 shows the layer-wise shrinkage of sintered samples, the green specimen diameter is taken as the initial value and the diameter of the specimens after sintering is

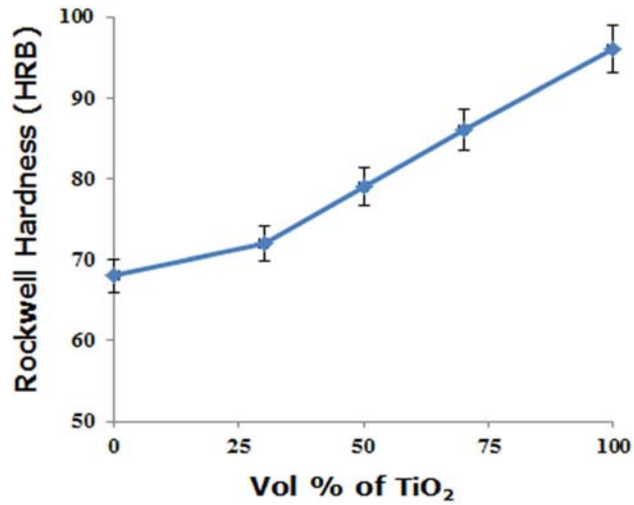

Figure 6. Hardness of 5 layered Ni/TiO_2 FGM.

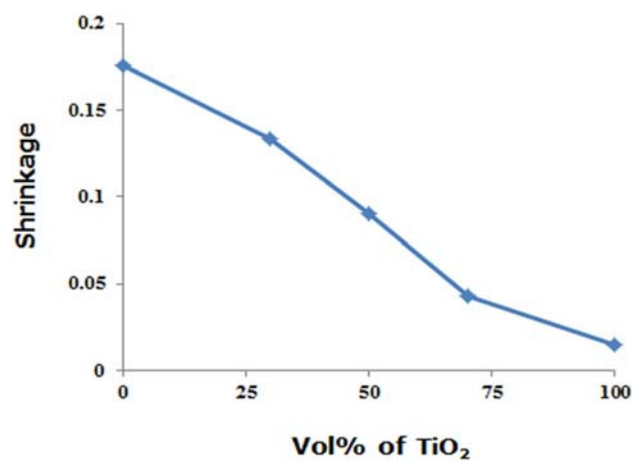

Figure 7. Shrinkage of 5 layered Ni/TiO_2 FGM.

measured. The increment of 0.1 mm in green sample diameter is noticed after compacting process. The results are mentioned in table 1 and the maximum shrinkage is observed in metal rich regions and decreases with increase of ceramic percentage.

4.5 *Compression test*

The compression test is conducted to know the stress-strain behaviour of sintered specimen. Several methods are proposed by the various researchers for determining the compression properties of metal matrix composite materials, but no method has been formally approved by ASTM for determining compressive properties of metal matrix composites [15]. Universal testing machine is used for the test;

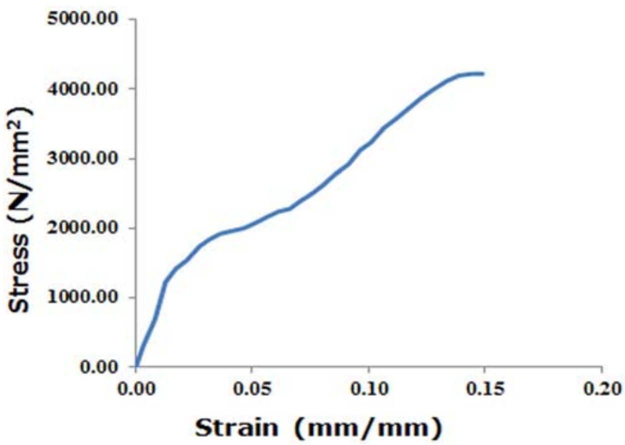

Figure 8. Stress-strain curve of 5 layered Ni/TiO_2 FGM.

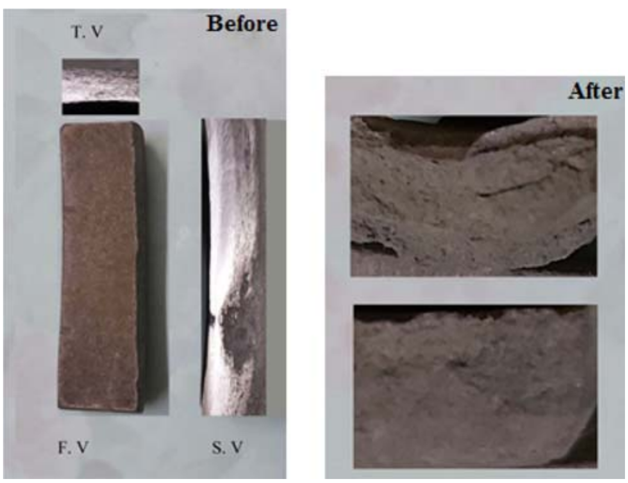

Figure 9. Photographs of compression test specimen.

the stress-strain behaviour of the sample is shown in figure 8. From the stress-strain graph the maximum stress in the specimen seen is 4220 N/mm^2. The samples are prepared as per ASTM D-695 standards and the photographs of the test specimen before and after the compression test is shown in figure 9.

5. Conclusions

The five layered Ni/TiO_2 FGM has been fabricated with the help of pressureless sintering technique and the SEM is used to observe the microstructure of the sintered specimen. The densification, distribution of ceramic reinforcement in metal phase and intermediate layers of the specimens are studied. As a part of mechanical characterization, the green and sintered densities of each layer of the specimen are calculated. The compositional variation of Ni/TiO_2 system and densities of the layers strongly depends on compacting process and sintering cycle. Cooling rate of the sintering cycle is attributed to be the reason for the thermal cracking in the ceramic rich regions. Rockwell hardness for each layer is measured and hardness increases with TiO_2 percentage. Linear Shrinkage of the specimen is measured and it increases with increase of Ni percentage. Compression test is conducted on the sintered specimen and stress- strain behaviour of the FGM is observed. The maximum stress in the specimen is 4220 N/mm^2 which is measured through compression test.

References

[1] Jin G, Takeuchi M, Honda S, Nishikawa T and Awaji H 2005 Properties of multilayered mullite/Mo functionally graded materials fabricated by powder metallurgy processing. *Mater. Chem. Phys.* 89: 238–243

[2] Birman V and Byrd L W 2007 Modeling and analysis of functionally graded materials and structures. *Appl. Mech.* 60:195

[3] Shen HS 2009 Functionally graded materials nonlinear analysis of plates and shells, Boca Raton: CRC Press

[4] Marin L 2005 Numerical solution of the Cauchy problem for steady-state heat transfer in two dimensional functionally graded materials. *Int. J. Solids Struct*. 42: 4338–4351

[5] Shahrjerdi A, Mustapha F, Bayat M, Sapuan S M and Majid D L A 2011 Fabrication of functionally graded hydroxyapatite-titanium by applying optimal sintering procedure and powder metallurgy. *Int. J. Phys. Sci.* 6(9): 2258–2267

[6] Gupta N, Prasad W, Madhu V and Basu B 2012 Ballistic studies on TiB2-Ti functionally graded Armor ceramics. *Def. Sci. J.* 62(6): 382–389

[7] Mueller E, Drašar Č, Schilz J and Kaysser WA 2003 Functionally graded materials for sensor and energy applications. *Mater. Sci. Eng. A* 362: 17–30

[8] Niino M, Kisara K and Mori M 2005 Feasibility study of FGM technology in space solar power systems (SPSS). *Mater. Sci. Forum* 492: 163–168

[9] Malinina M, Sammi T and Gasik M M 2005 Corrosion resistance of homogeneous and FGM coatings. *Mater. Sci. Forum* 492–493: 305–310

[10] Erikssona M, Mohamed R and Shena Z 2013 Spark plasma sintering of WC, cemented carbide and functional graded materials. *Int. J. Refract. Met. Hard Mater.* 36: 31–37

[11] Garciaa J and Pitonakb R 2013 The role of cemented carbide functionally graded outer-layers on the wear performance of coated cutting tools. *Int. J. Refract. Met. Hard Mater.* 36: 52–59

[12] Gillia O and Caillens B 2011 Fabrication of a material with composition gradient for metal/ceramic assembly. *Powder Technol.* 208: 355–366

[13] Traini T, Mangano C, Sammons R L, Mangano F, Macchi A and Piattelli A 2008 Direct laser metal sintering as a new approach to fabrication of an isoelastic functionally graded

material for manufacture of porous titanium dental implants. *Dental Mater.* 2(4): 1525–1533
[14] Durejko T, Lipiński S, Bojar Z and Bystrzycki J 2011 Processing and characterization of graded metal/intermetallic materials: the example of Fe/FeAl intermetallics. *Mater. Des.* 32: 2827–2834
[15] John W S 1988 Metal matrix composites testing, analysis and failure modes. ASTM STP: 1032, Philadelphia, pp. 7–18