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Microstructure and Microhardness of ZrO_2 Reinforced PM 316L Austenitic Stainless Steel Composites Sintered in Ambient and Argon Atmosphere

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Abstract: ***The present study examines the microstructure and microhardness of zirconia (ZrO_2) reinforced PM 316L austenitic stainless steel matrix composites. ZrO_2 was added in a proportion of 1 wt% to 3 wt%. Powders were compacted into a disc of 11mm diameter and 2mm thick at 70kN. Compacts were sintered in an ambient and argon atmosphere at 1250°C for 30 minutes. Sintered compacts were then analyzed for microhardness using Vickers hardness testing machine, and microstructure was examined using a scanning electron microscope. The study revealed that the reinforcement of ZrO_2 significantly enhanced the microhardness of PM 316L SS matrix composites with a microstructure consisting of irregular porosity and zirconia encapsulating the 316L SS particles.***

***Index Terms*: Microstructure, Microhardness, Powder Metallurgy, 316L Austenitic Stainless Steel Composites.**

I. INTRODUCTION

A composite material is an amalgamation of at least two physically and chemically distinct materials, one being the continuous phase called matrix and the dispersed phase called reinforcement, the resultant material possesses characteristics different from the individual phase. Classification of composites is generally based on the type of matrix used; a major group of composites includes metal matrix composites (MMC), polymer matrix composites (PMC), ceramic matrix composites (CMC), etc.

In recent times, extensive research is focused on MMCs due to their increased demand in a high-performance application. MMCs are composites having metal or alloy matrix, reinforced with ceramics in the form of fiber, particulate, or whisker [1]. MMCs are manufactured by various methods like liquid state method, solid-state method, semi-solid state method, vapor deposition, etc. Powder metallurgy (PM) is a solid-state technique of synthesizing MMCs, where the matrix metal powder and the discontinuous phase are mixed and then bonded through a process of compaction and sintering. With powder metallurgy simplest to intricate components can be produced and composition can be altered and studied as per the requirement. Stainless steel composites form one of the major groups of MMCs produced through powder metallurgy route. Apart from promising mechanical properties, 316L stainless steel exhibits excellent corrosion resistance due to the presence of chromium, nickel, and molybdenum. PM 316L stainless steel finds a wide range of applications including agricultural, aerospace, biomedical, chemical, nuclear plants, etc. [2 -5]. Sintered PM 316L stainless steel matrix composites can be obtained from stainless steels powders by the simple addition of metal oxides, followed by compaction and sintering process. [6]

I. Sulima et al. reported that the TiB_2 reinforced in stainless matrix tend to occupy the sites along the grain boundary forming fine precipitates of complex borides with chromium. Size of boride was affected by the SPS method applied to sinter the composites. Increased hardness was observed with the increase in TiB_2 content, for composite with 8% TiB_2 they obtained the hardness as high as 265 GPa which is nearly twofold the pure 316L steel sintered compact [7].

The microstructure of steel composite reinforced with TiC produced isolated pores contrary to the pure samples that showed interconnected pores features. The concentration of reinforced particles at grain boundaries increased with the amount of TiC. Reinforcement of TiC had a positive effect over the microhardness of steel composites which increase from 290 to 380 HV0.01 [8].

Stainless steel composites with a uniform dispersion of Al_2O_3 particles in steel matrix along the grain boundaries were produced by R. Tongsri et al [9]. Addition of Al_2O_3 had a proportionate effect over the hardness of the composite which can be attributed to the hardness and resistance of reinforced particle to deformation of steel matrix during hardness test. According to the investigation by Patel et al. [10], the microstructure of sintered PM 316L stainless steel reinforced with low melting point oxide particles exhibit reduced porosity as compared to the steel composites reinforced with oxide particles having a high melting point.

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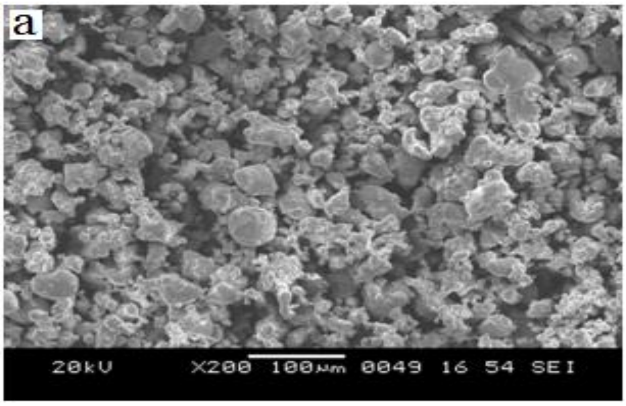
Incorporation of these hard particles significantly resists the deformation of matrix and consequently increased the hardness of 316L steel composites. Increased proportion of 3.5 wt% MgO partially stabilized zirconia powder (MgPSZ) reinforced in metastable austenitic steel powder ceases the grain growth resulting in a fine grain structure. Fine MgPSZ particle accommodates in the intergranular spaces to form clusters. Up to 5% volume fraction of MgPSZ resulted in increased strength as compared to pure steel. The rise in strength could most probably be due to the fine-grained structure of the matrix [11]. In order to improve the environment resistant surface, M.Atik et al. [12] used zirconia as a coating material over 316L steel due to its thermal resistance to oxidation, ionic conductivity, alkali resistance, and the mechanical properties. Zirconia as reinforcement in 316L stainless steel has not been reported yet.

The sintering atmosphere also forms an important factor that influences the microstructure and properties of PM steel. Vacuum and gasses like hydrogen, nitrogen, argon, etc have been reported as sintering atmosphere. The stainless steel sintered in argon atmosphere achieved enhanced densification as compared to samples sintered in an N_2 atmosphere. The nitrogen from sintering atmosphere forms Cr_2N which hinders the diffusion rate thereby reducing the sintered density [13]. N. Kurgan [14] reported globular pores in the steel samples sintered in a nitrogen atmosphere in contrast to the samples sintered in argon atmosphere which achieved high pore ratio and irregular pore profile. Samples sintered in nitrogen atmosphere tend to become more harder than those sintered in an argon atmosphere, this can be attributed to the solid solution strengthening effect of nitrogen which diffuses in the samples during sintering. Sintering in the ambient or oxygen-rich atmosphere has not been reported yet [15].

The purpose of present work was to study the evolution of microstructure and evaluate microhardness of PM 316L austenitic stainless pure and composites reinforced with ZrO_2, sintered in an ambient and inert atmosphere.

II. MATERIALS AND PROCESS DESCRIPTION

The matrix material used was water atomized 316L austenitic stainless steel powder. The SEM image (Fig.1) reveals the presence of smaller (5µm) and larger (50µm) particles, the mean of particle size calculated was around 45µm. The shape of the particles is found to be irregular which is the characteristic of water atomization process.



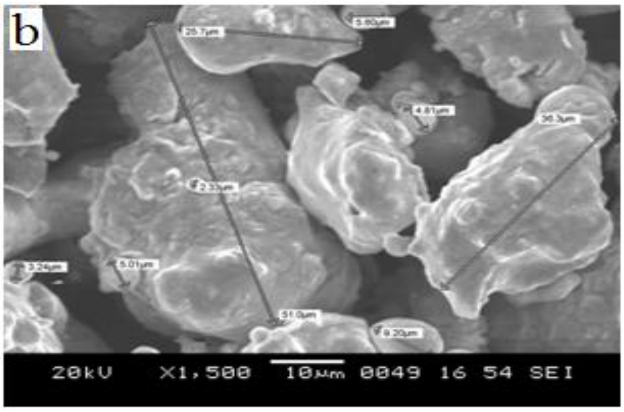


Fig.1. SEM micrographs of 316L SS powder (a) x200 and (b) x1,500

Table.1
Characteristics of 316L SS and ZrO_2

	Specifications	316L	ZrO_2
1	Density (g/cm³)	7.99	5.68
2	Melting Point (°C)	1399	2715
3	Crystal Structure	FCC	Monoclinic below 1170 °C, tetragonal between 1170 °C to 2370 °C, and cubic above 2370 °C [13]

The ZrO_2 powder having an average particle size of 5 µm was used as the reinforcement. Comparative characteristics of 316L SS and ZrO_2 powder are listed in Table.1. The ZrO_2 powder was added in a proportion of 1-3 wt% to 316L austenitic stainless steel powder and was manually blended using mortar and pestle for 20 minutes. The blended powders were uniaxially compacted at 70kN using a hydraulic press. Zinc stearate was used as die wall lubricant to minimize the friction and facilitate the smooth ejection of the compacted pellet. The diameter and thickness of compacted pellets were 11 mm and 2 mm respectively (Fig.2). The green compacts were sintered in a muffle furnace at 1250°C for 30 minutes in an ambient and argon atmosphere. The elemental composition of PM 316L stainless steel used in this work is given in Table.2.

Fig.2. Sintered Pellet

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Table.2
Composition of 316L austenitic stainless steel

Elements	Cr	Ni	Mo	Mn	C	Si	S	P	Fe
W%	17.47	12.52	2.25	0.33	0.03	0.75	0.028	0.04	Balance

The microstructure of finely polished sintered compacts was examined by scanning electron microscope (model: JED 2300, JEOL, Japan). Microhardness of sintered samples was determined by Vickers hardness tester at 0.3 kg load. The observed hardness values are the average of five readings taken at random spots throughout the sample.

III. RESULTS AND DISCUSSION

The SEM micrographs of finely polished pure 316L SS compacts sintered in ambient (PAM) and argon (PAR) atmosphere, at the magnification of 1000x are shown in the fig 3.a and 3.b respectively.

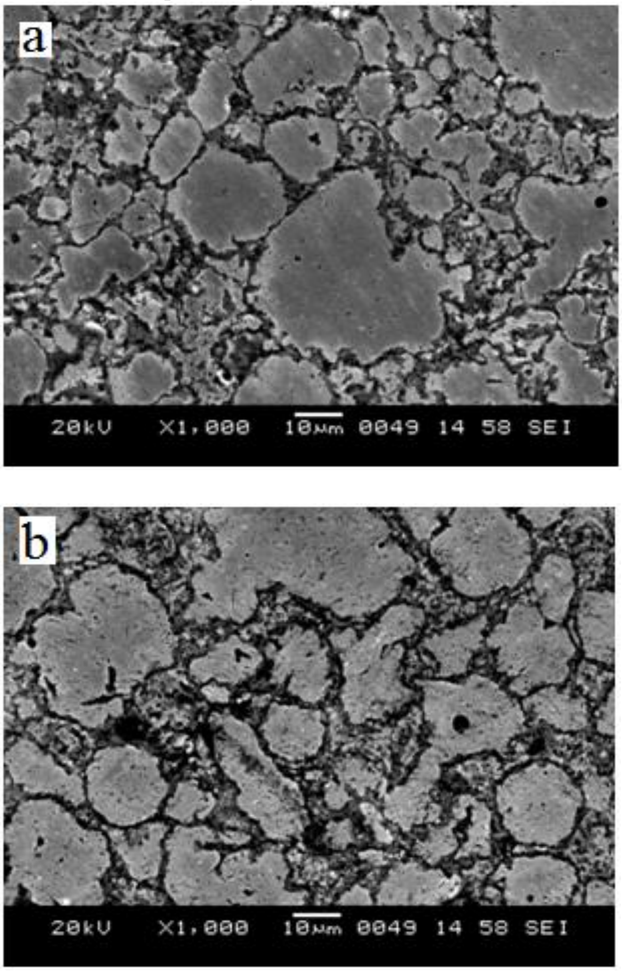

Fig. 3. SEM micrographs of plain 316L compacts sintered in (a) ambient and (b) argon atmosphere

The inter-particle regions appear to be less porous in PAM as compared to PAR. The pores are irregular, randomly distributed over the visible surface and reveal a tendency to connect with each other. Some of the small pores are also visible within the particles that were formed during powder production. It has been reported that the water atomized steel powders have high oxygen content due to the thick oxide layer formed on the particle surface during production [16, 17]. According to Y. Hedberg et al. [18] the surface of water atomized steel particles are strongly enriched with oxidized Si (silicates).

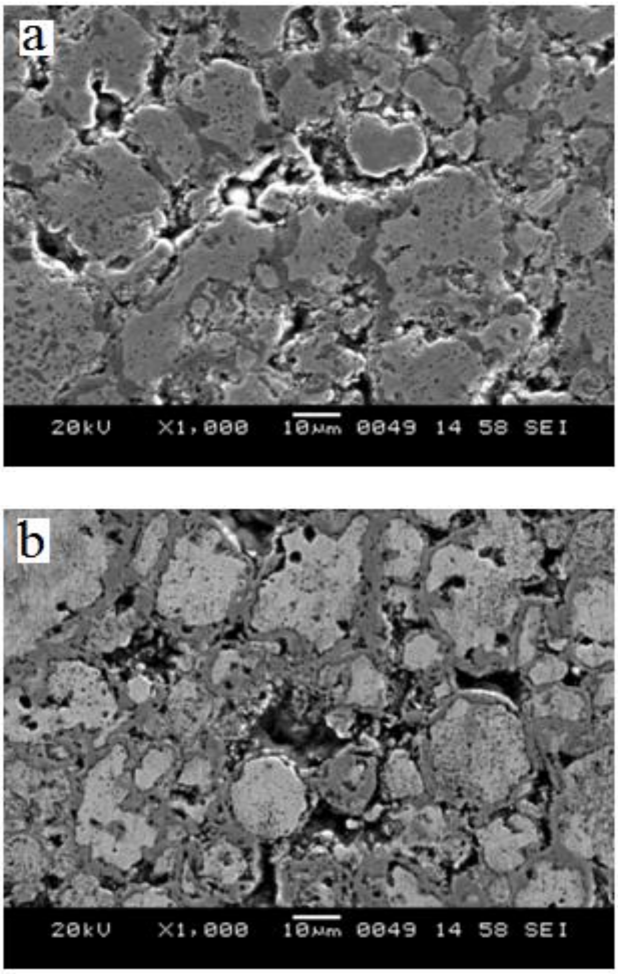

Fig. 4. SEM micrographs of 316L+3% ZrO_2 compacts sintered in (a) ambient and (b) argon atmosphere

Based on these fact it is assumed that the oxide layer on the surface of the particles tends to become more active in argon atmosphere inducing the dissociation of the same at the sintering temperature, resulting in higher irregular interconnected porosity at inter-particle regions in PAR. The particles are found to shrink in case of PAR which can be a possible reason for its increased hardness of PAR (Table 3). Another possible reason for the increased hardness of PAR could be the solid solution strengthening promoted by argon atmosphere as reported by M. Dewidar [15]. Steel composites reinforced with zirconia reveals distinct microstructural features when sintered in ambient (ZAM) and argon atmosphere (ZAR), Fig 4a and 4b are SEM micrographs of sintered ZAM and ZAR respectively. Ceramic reinforcement inhibits the grain growth, resulting in a slight decrease in average grain size [11]. In both the cases, the ZrO_2 is found to accumulate at the inter-particle regions and encapsulating the steel particles.

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Zirconia in ZAM exhibits bonding with steel particles whereas in ZAR it assumes and retains the particle surface shape but it dissociates from the particle surface due to the shrinking of steel particles. Excess ZrO_2 filling the larger inter-particle space doesn't sinter at 1250°C, as is obvious from its high melting point, causing weak ceramic particle interface, therefore it leads the higher porosity in sintered ZAM and ZAR compacts. Pore number and size continue to rise with the further addition of ZrO_2.

Table.3 and 4 shows the average Vickers hardness values of compacts sintered in ambient and argon atmosphere respectively. Bar graph (Fig.5) compares these values. The hardness of steel composites is found to increase with zirconia reinforcement.

Table. 3 Microhardness of 316L- ZrO_2 composites sintered in ambient atmosphere

Sample	Wt% ZrO_2	HV
PAM	0 % ZrO_2	246
1ZAM	1 % ZrO_2	426
2ZAM	2 % ZrO_2	309
3ZAM	3 % ZrO_2	355

Table. 4 Microhardness of 316L- ZrO_2 composites sintered in argon atmosphere

Sample	Wt% ZrO_2	HV
PAR	0 % ZrO_2	292
1ZAR	1 % ZrO_2	431
2ZAR	2 % ZrO_2	366
3ZAR	3 % ZrO_2	432

In order to understand this, it is first necessary to understand the behavior of ZrO2 at various temperatures. ZrO2 undergoes allotropic transformation at different temperatures viz monoclinic up to 1170 °C, tetragonal between 1170 °C to 2370 °C, and cubic above 2370 °C [20].

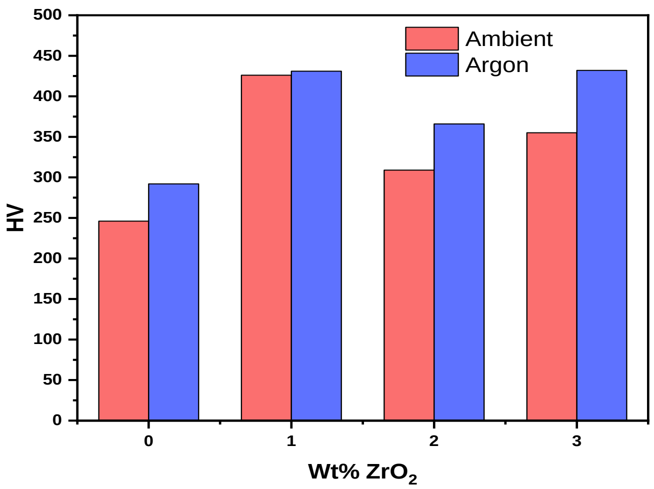

Fig. 5. Effect of ZrO_2 and atmosphere on microhardness.

This allotropic transformation of ZrO_2 from tetragonal at high temperature to monoclinic during cooling causes volume expansion of 3%-5%, which induces compressive stress in the surrounding steel matrix [20,21]. Grain growth is inhibited by the increased ceramic reinforcement which may also be contributing to the enhanced microhardness of the composites. The hardness of ZAR is found to be higher than ZAM this could be due to the shrinking of particles favored by argon atmosphere.

IV. CONCLUSION

From the present work, we conclude the following.

1. Sintering in argon atmosphere leads to more porous particle interface both in pure as well as ZrO_2 reinforced composites in contrast to those sintered in the ambient atmosphere.
2. ZrO_2 reinforced in the matrix tends to encapsulate the 316L particles. Bonding between ZrO_2 and 316L particles was observed in ZAM.
3. Composites sintered in argon atmosphere tend to exhibit high microhardness as compared to the samples sintered in the ambient atmosphere.
4. Among all the sintered samples, composites with 3% ZrO_2 sintered in argon atmosphere achieved the highest microhardness.

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