Spark Plasma Sintering of 2507 Duplex Stainless Steel Reinforced with TiC

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Abstract. As technological development is advancing towards the use of powder metallurgical (PM) processed stainless steels for automotive and structural applications, 2507 duplex stainless steel have gained considerable scientific attention and technological interest due to potential benefit associated with their unique properties such as corrosion and oxidation resistance and good formability. However, application of this material is hindered by its low mechanical strength and poor anti-friction properties resulting from the elongated porosity which acts as stress concentration sites that could lead to premature and brittle failure at a relatively lower load. These drawbacks can be improved using appropriate technology. Effort was made in this study to reinforce 2507 stainless steel with TiC particles and consolidate with spark plasma sintering (SPS). A relative density of 99.7% and Vickers hardness of 289.7 HV was obtained for 2507 DSS sintered at 1000 ^{o}C. The hardness value of 2507 stainless steel containing 10 vol%TiC was found to be 296.03 HV. The microstructure of the material produced was investigated using SEM.

1. Introduction

Technological developments of advanced materials for better industrial applications are ongoing. Powder metallurgy is one of the methods used in synthesizing these material composites as in processed steels for automotive and structural applications [1,2]. Powder metallurgy gives good densification, low cost net-shaping, relatively low-temperature processing, high material utilization (95%) and the ability to tailor microstructures for specific applications [3].

One such material, 2507 duplex stainless steel, has gained considerable scientific attention and technological interest due to its unique properties such as corrosion resistance, oxidation resistance and good formability. These properties are attributed to the relative high Cr (25 wt%), Ni (7 wt%) and Mo (4 wt%) contents as compared to other steels [4]. Typically, 2507 duplex stainless steel finds applications in hostile environment such as in desalination plants, oilrig structures, firefighting systems, chemical process industries, among others [4]. The Fe component has a mixed microstructure of austenite and ferrite in 50/50 ratio.

However, Duplex 2507 stainless steel, in temperatures above 250 °C, forms intermetallic phases, becomes brittle and compromises its useful mechanical and physical properties such as corrosion resistance and hardness [5]. In other to improve duplex 2507 properties, and possibly enhance its temperature limitation, various attempts such as adding ceramics particulates have been undertaken [6, 7]. Among the materials of choice, Titanium Carbide (TiC), Titanium Nitride (TiN) and Titanium Carbo-Nitride (TiCN) have emerged as good reinforcements to austenitic stainless steels due to their excellent properties - high hardness, low density, high melting temperature, high modulus, excellent wear and corrosion resistance, good wettability and stability in steel melt [8]. In addition, TiC particles are thermodynamically stable in an iron alloy matrix at the low sintering temperatures, with practically negligible (0.5–1%) solubility [9]. This limited solubility might have resulted in the formation of the (Fe, Cr)-rich precipitates at the interface which contributed to the improved strength of the component [9]. However, homogeneous mixing and fabrication of ceramics and steel powders into useful material composite has been daunting task due to surface contamination of impurities [10] and the creation of unwanted pores that affect densification [7] of the final composite product [1].

Lin and Xiong [11] investigated the microstructure and mechanical properties of 316L stainless reinforced with TiC prepared by microwave sintering and warm compaction. They reported that the relative density, hardness and abrasive properties of 316L stainless steel could be improved by using 5 wt.% TiC particles as reinforcements in composites towards a maximum relative density of 94.8%.

Spark Plasma Sintering (SPS) brings some exceptional advantages into the sintering of materials due to its unique features [12]. The accurate control of sintering temperature as well as high temperature up ramp speed makes SPS a promising technique for producing highly dense materials with controlled grain growth [12]. Li *et al.* [12] reported on rapid fabrication of *in-situ* TiC particulates reinforced Fe-based composites by spark plasma sintering. They obtained a maximum relative density of 99.2% at the following sintering consolidation: applied heating rate from room temperature was about 80 °C/min until sintering temperature of 1150 ^{o}C, holding time and pressure of 5 minutes and 30 MPa in vacuum, respectively.

In the present study, attempt to reinforce 2507 duplex stainless steel powder of average size 3 – 40 μm with TiC powder (2 - 15 μm) of different compositions (10, 15 and 20 vol.%) was undertaken. The objectives were to check for good sinteribilty, densification and hardness of the composites. Two sintering consolidation temperatures used were 1000 ^{o}C and 1100 ^{o}C.

2. Materials and methods

2.1. Feed stock powder and characterization

Dry elemental mixing of the powders were performed with 3 stainless steel balls (9.5 mm diameter) in a Turbula T2F mixer at a rotating speed of 72 rpm for a period of 4 hours. The High Resolution Scanning Electron Microscope (HRSEM Joel 7600F) equipped with Energy Dispersion X-ray (EDX) was used to check the homogeneity of the powder mixture.

2.2. Consolidation and characterization of sintered samples

The powder mixtures were consolidated by Spark Plasma Sintering (HPD 25, FCT System GmbH, Germany) furnace. The powders were loaded into a graphite die 30 mm diameter and a sintered sample thickness obtained was 5 mm. A minimum load of 10 kN (14.1 MPa) was applied to establish good electrical contact between the powder particles and the die assembly. The consolidation was carried out under a vacuum of 0.56 mbar. The mixed powders were uniaxial pressed at 50 MPa.

2.3. Characterization of the sintered samples

The densities were measured using the Archimedes's principle method. Samples for analysis were cut in a plane perpendicular to the pressing direction, hot mounted and grinded using different sizes of silicon carbide paper ranges from 120 to 1200 grit. The samples were then polished using 3 μm diamond suspensions on a polishing cloth for 5 minutes at a speed of 300 rpm. The final polishing was done with 1 μm diamond paste. The microstructure investigation was performed on the polished surface using HRSEM equipped with EDS analysis. Microhardness measurements were made on the as-polished specimens using the Vickers microhardness tester at 100 g load for 15 s. The samples' surfaces were indented randomly at five different positions for each sample and the average hardness values were recorded. The fracture surface of 2507 stainless steel containing TiC was analysed to evaluate the sinterability of material and TiC bonding into the matrix.

3. Results and discussion

Figure 1 shows the SEM micrograph of 2507 stainless steel powder particles. The starting powders revealed spherical shape and agglomerates of smaller particles. The micrograph also shows that the powder particles size ranged from 3 μm to 40 μm.

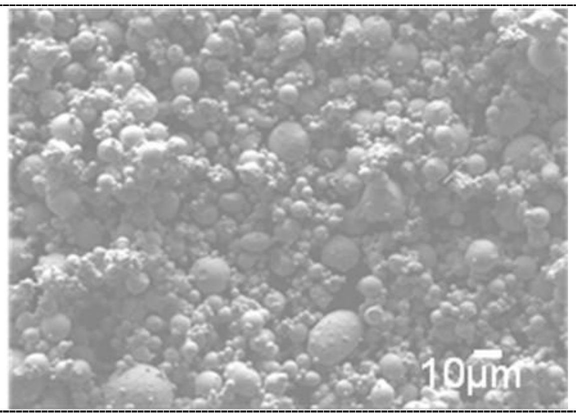

Figure 1. SEM micrograph of 2507 stainless steel powder

Figure 2 shows the SEM micrograph of the as-received TiC powder particles. The particles were jagged and large fractured irregular surface. Also seen in the micrograph are inherent pores created because of the high reaction heat released during the powders' formulation.

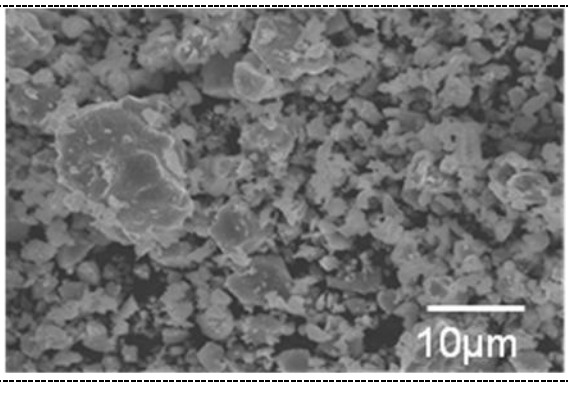

Figure 2. SEM micrograph of TiC powder

Sintering of crystalline materials can occur by several mechanisms such as surface diffusion, lattice diffusion, grain boundary diffusion and dislocation motion. All these processes are temperature dependent. An increase in temperature would cause the diffusion of atoms and this would results in necking as well as shrinkage (densification) [13]. Table 1 summarizes the relative density of the neat 2507 stainless steel and that with the TiC composites. A relative density of 99.7% was obtain for as-received – neat - 2507 sample at both sintered temperatures at 1000 ^{o}C and 1100 ^{o}C. The high densification could be ascribed to the spherical shape and size as well as the enhance activation of plasma which resulted in large quantity of necking and welding of constituents [14]. Good sintering was therefore achieved for the neat 2507 stainless steel.

The increasing content of TiC particles reduced the relative density of 2507 DSS/TiC composite samples, sintered at both temperatures. The reduction was due to the low specific weight of the TiC particles [7] and the formation of micro-voids in the constituents (spherical dimples - see figure 4). However, for the same composite, increase in temperature, produced higher densification because of grain size expansion and the reduction of pore volume/void size.

Table 1. Results of the relative densities and microhardness of sintered sample

Sample vol.%	Consolidation condition (temperature) at 50 MPa, heating ramp rate 100^{o}C/min and holding time of 5 min	Density (%)	Microhardness (HV)
2507 DSS	1000^{o}C	99.7	289.7±0.2
2507-10TiC	1000^{o}C	97.3	281.7±0.5
2507-15TiC	1000^{o}C	95.5	266.9±0.7
2507-20TiC	1000^{o}C	93.2	259.1±1.2
2507 DSS	1100^{o}C	99.7	289.7±0.2
2507-10TiC	1100^{o}C	98.3	296.3±0.3
2507-15TiC	1100^{o}C	97.6	281.4±0.6
2507-20TiC	1100^{o}C	94.7	279.9±0.8

Figure 3 shows the SEM micrograph of 2507 stainless steel matrix reinforced with 20 vol% TiC sintered at 1100 ^{o}C with holding time of 5 minutes. The image shows the composites have a uniform distribution of the hard but less dense TiC (dark patches in Figure 3) within the matrix without cracks.

The measured microhardness of the neat 2507 stainless steel samples was 289.7±0.2 HV. The value did not change for the sintered temperatures at 1000 and 1100 ^{o}C. Measured microhardness value, however, decreased with TiC content additions at a particular sintering temperature; and this could be due to the reduction in their densities value. This is contrary to Lin and Xiong [10] reported result. They found that the hardness of composites of TiC in 316L stainless steel increase with increasing TiC content. Possible reason for the present result could be due to the jagged shape of the TiC powder and the sintering temperatures used. Clearly, (see the indicated 'inert' TiC in Figure 4) there was not adequate sintering of the composites at the chosen consolidation parameters – temperatures, uniaxial pressure and dwell time – that should have been increased. However, the hardness value of the composite matrix with addition of 10 vol.% TiC, increased from 281.7 (at 1000 ^{o}C) to 296.3 HV (at 1100 ^{o}C). This indicated 1100 ^{o}C to be better sintering temperature than 1000 ^{o}C. Furthermore, measured values of the other composites also showed about 5% (15 vol% TiC) to 8% (20 vol% TiC) microhardness increase from sintering temperatures of 1000 to 1100 ^{o}C. The increased hardness is attributed to possible segregation of impurity elements (N and C) from the bulk to the surface resulting in the formation of harder nitrides and carbides.

Figure 4 shows the fractograph of 2507 stainless steel reinforced with 10 vol% TiC sintered at 1100 ^{o}C and hold for 5 min. The fractograph reveals dimple structure – a sign that reveals microvoids in the intra- and inter-bonding of the components. Some sections of the figure also show physical (other than metallurgical) bonding between the matrix and TiC indicating incomplete sintering at 1100 °C.

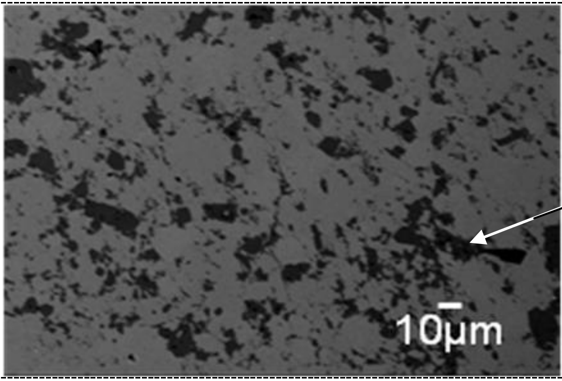

Figure 3. Distribution of 20%TiC in 2507 DSS at 1100 °C

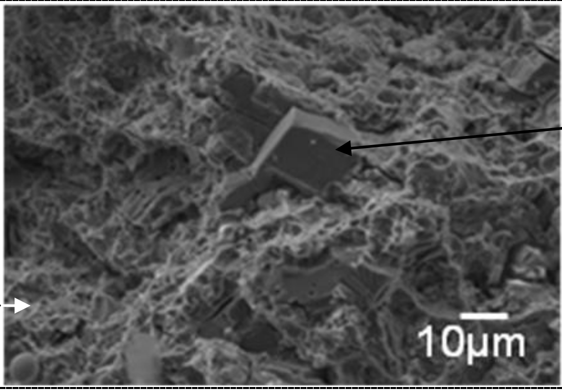

Figure 4. Fractopgraph of 10%TiC in DSS composites sintered at 1100 °C

4. Conclusion

A processing technique was developed to incorporate different TiC compositions homogeneously into the 2507 stainless steel matrix and consolidated by Spark Plasma Sintering. Composite hardness does not just increase with increasing TiC content but depends on factors such as sintering temperature, powder size and shape, composite densification and perhaps a critical vol.% of the TiC in the matrix. The 2507 DSS-10 vol.%TiC consolidated at 1100°C was found the hardest (296.3 HV) and best dense composite (relative density of 98.3%).

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