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EFFECT OF THE α-Si_3N_4 ADDITION ON THE TRIBOLOGICAL PROPERTIES OF 316L STAINLESS STEEL PREPARED BY ATTRITION MILLING AND SPARK PLASMA SINTERING

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Abstract

The submicron sized α-Si_3N_4 has been used to study the effect of ceramic addition on the tribological properties of the commercial 316L stainless steel (SS) prepared by powder metallurgy. Two samples consisting of 316L SS (reference) and 316L with 1wt% Si_3N_4 have been prepared using attrition milling and Spark Plasma Sintering (SPS). The density, hardness and friction coefficient of reference and the composite have been investigated and are presented in comparison with literature results.

1. Introduction

Friction is the natural response of all tribosystems [1]. The fabrication process has great influence on the microstructure. This last property has great and direct influence on the tribological properties of the material. *J. Menga et al* investigated the tribological properties of the 316L made by Powder Injection Molding (PIM) sliding against AISI 52100 steel at low loads [2]. The formation of carbide phase during the sintering in vacuum improved the tribological properties of the stainless steel, where a lower wear rate and friction coefficient have been recorded. The wear mechanism has been influenced by a combination of the formation of tribo-layer and various iron oxides. In case of the sintered samples in hydrogen, severe worn marks have been observed on the surface [2]. *F. Bartolomeu et al* found that the 316L stainless steel made by Selective Laser Melting (SLM) shows better tribological properties in comparison to the conventional casting and hot pressing because of its finer microstructure [3]. For this, SLM is a promising process to produce 316L SS with improved wear resistance. *Y. Li et al* studied the tribological behavior of the oxynitrided austenitic stainless steel produced by Active Screen Plasma (ASP) [4]. They have found 95% decrease of the wear rate in the case of the treated samples, where abrasion marks and oxidation have been slightly observed on the worn surface. In the case of untreated samples a plastic deformation and intense adhesive wear have been observed. *D. Guan et al* investigated the polycarbosilane (PCS) reinforced 316L made by Spark Plasma Sintering (SPS) [5]. In their

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study the 3% PCS samples show the best wear resistance, where 86% decrease of the wear rate have been observed comparing to the pure 316L. In the same time higher microhardness values have been observed with the increase of PCS content.

P.F. Wang et al investigated the tribological properties of a Gradient Nano-Grained (GNG) layer fabricated by Surface Mechanical Rolling Treatment (SMRT) on a 316L S [6]. The sliding tests have been performed at room temperature in dry and oil lubricated conditions where it found that the GNG layer enhanced the wear resistance in both cases. *Y. Wang et al* studied the effects of surface nanocrystallization on tribological properties of 316L stainless steel by ultrasonic cold forging technology (UCFT) under different lubrication conditions [7]. It has been found that the low friction and antiwear performances of samples are influenced by the synergistic effects of the surface nano crystallization. *F. Rotundo et al* investigated the low temperature carburizing (LTC) influence on dry sliding tribological behavior of the 316L stainless steel at high and room temperature [8]. The LTC increased the hardness of the 316L significantly which in turn improved the tribological properties of the steel when tested at room temperature. In the case of high temperature testing, the tribological behavior of steel becomes similar to that of an untreated steels. .This can be explained by the structural changes in the carburized layer due the high temperature combined with friction heating, and the formation of an oxide protective layer in the case of non-treated 316L SS.

In present study the effect of the addition of the submicron sized α-Si_3N_4 to commercial 316L stainless steel processed by attrition milling and spark plasma sintering has been investigated. The density, hardness and friction coefficient of both samples have been determined and compared with recent reference data.

2. Experimental Procedure

The submicron sized α-Si_3N_4 powder (UBE) has been added to the commercial 316L SS powder (Höganäs) to prepare a Ceramic Dispersion Strengthened Steel (CDS) with the following composition 316L/1wt% Si_3N_4. The starting globular 316L SS powder has an average grains size of ~70µm with the presence of satellites. The polygonal α-Si_3N_4 powder has an average grains size of ~200 - 300 nm. In order to reduce the grains size of steel powder and ensure a homogeneous distribution of the α-Si_3N_4 in the steel matrix the Union Process attritor mill type 01-HD/HDDM has been employed. The powders have been milled in ethanol for 5 hours at 600 rpm.

The two powder samples, one reference and one composite have been sintered in vacuum at 900°C under 50 MPa for 5 minutes (dwelling time) using the Sinter-SPS-7.40MK-VII apparatus, installed in Istanbul Technical University. The short time of sintering (30 minutes, including 5 minutes holding time) prevents the excessive grain growth. After sintering, the solid discs of ~100mm diameter and ~ 9mm thickness have been obtained. The solid discs have been cut into small bars for the mechanical properties testing.

The samples have been immersed in water for 3 days in order to measure their real density using Archimedes' principle. The samples hardness has been measured using Vickers method by applying 5 N load for 30 seconds.

The tribometer type CSM-HT-Tribometer has been used to investigate the tribological properties of sintered samples. Different grinding papers (up to 1000µm) have been used for polishing the samples before measuring the tribological properties. 5 N normal load was

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applied to load the 5mm diameter Si_3N_4 ball against the steel sample surface with 1mm shift from the rotation axe of the sample. The samples have been tested for 2161 m at room temperature in air (without lubricant) with ~49% atmosphere humidity. The scanning electron microscopy (SEM, Zeiss-SMT LEO 1540 XB and Jeol JSM-25-SIII) have been used to investigate the structures and morphologies of the milled powders and surface of sintered samples before and after the tribological investigation.

3. Results and discussion

After attrition milling, the presence of three different grain morphologies have been observed in both of milled samples, reference (316L) powder (Figure 1A) and the CDS powder (316L/1wt% Si_3N_4). A homogeneous distribution of the α-Si_3N_4 particles in the steel matrix was observed after intensive milling, more details were presented in our previous publication [10].

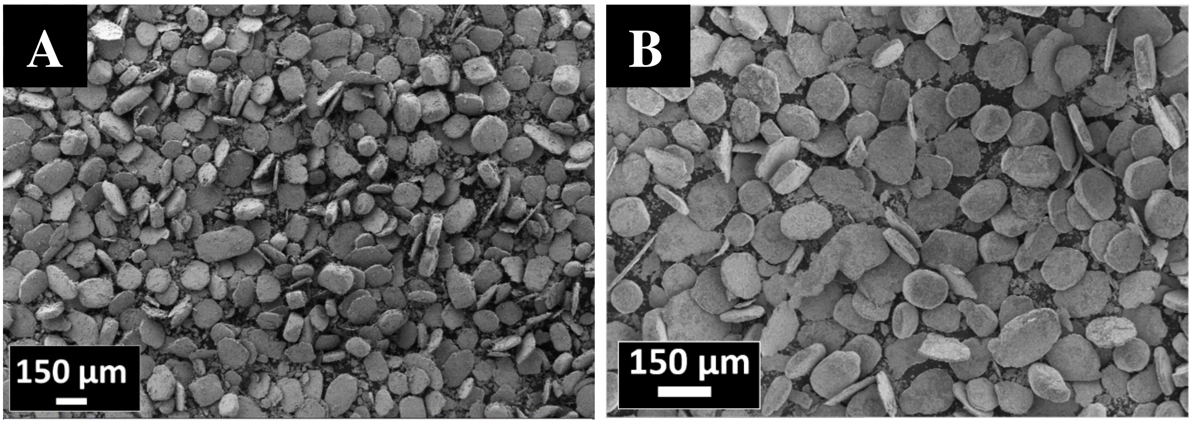

Fig. 1. *SEM images after attrition milling: A) 316L reference powder, B) 316L/ 1 wt % Si_3N_4.*

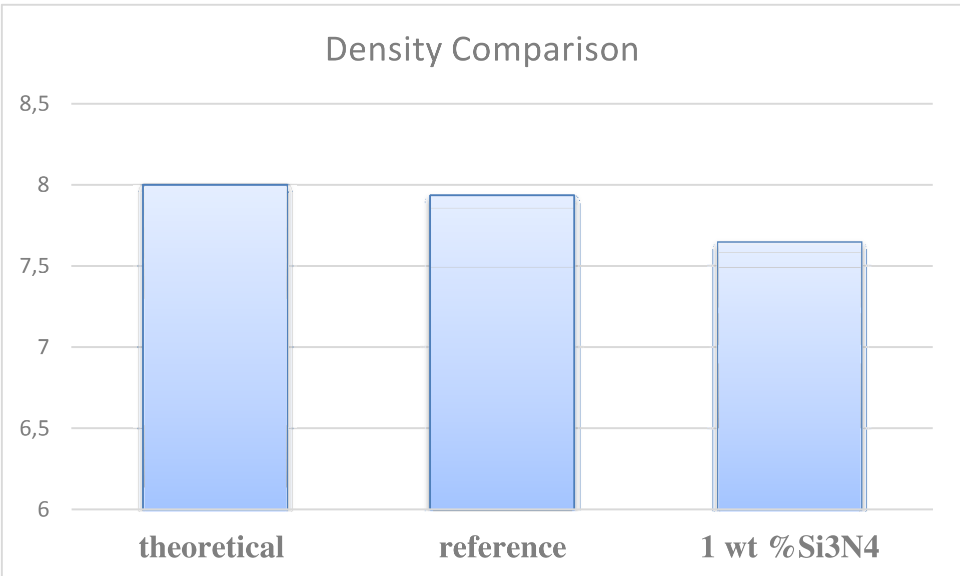

Fig. 2. *Comparison of the obtained densities with the steel theoretical density (g/cc)*

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Powder mixture and reference were sintered by SPS. The density results showed that the sintered reference sample had an average density of 7.93 g/cc. That means that in comparison with the theoretical density of the 316L steel (8 g/cc) the sintered reference sample had a relative density of 99.17% (Fig. 2). In the case of 316L/1wt% Si_3N_4 composite, 95.56 % relative density in comparison to theoretical density was measured. Density value of 7.64 g/cc was characteristic for this composite (Fig. 2).

The microstructural observations of the sintered reference confirmed the morphology of the base 316L SS. On the other hand, the presence of two different phases (Fig. 3B) was shown for sintered 316L/1wt% Si_3N_4 composite. The bright phase (Fig. 3B "1") showed a maximum hardness of 274.6 HV, where the dark phase (Fig. 3B "2") showed a higher hardness with 727.5 HV due to the higher amount of silicon nitride and silicon oxide [10].

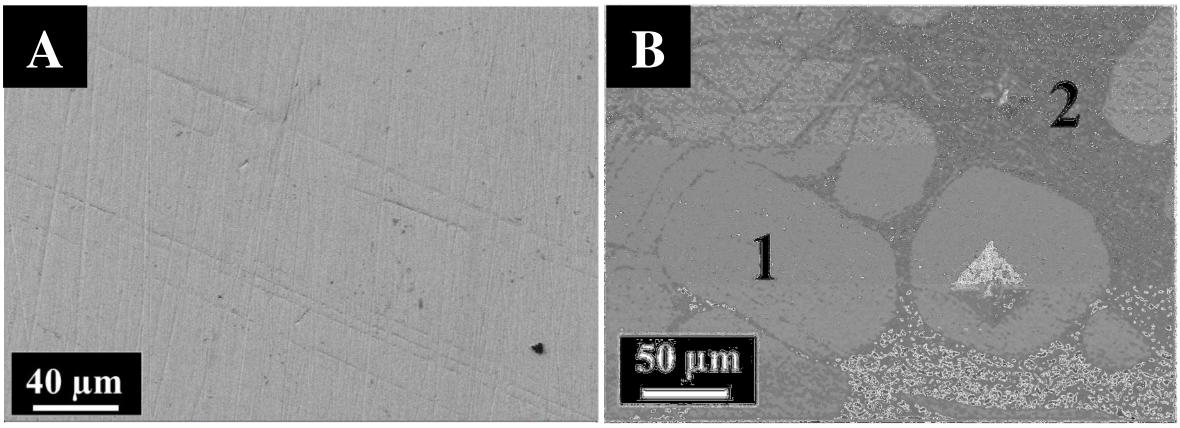

Fig. 3. *SEM investigations of A) sintered 316L reference sample, B) sintered 316L/1 wt Si_3N_4 composite with the hardness prints on different phases.*

The Fig. 4 shows the micro-hardness results of the elaborated composites in comparison with other hardness results of the 316L SS made by three different processing technologies [3]. The reference sample showed an average micro-hardness of 178.6 HV which is 8.24% higher than the 316L SS hardness produced by casting process, this is probably due to the finer microstructure maintained by the SPS process.

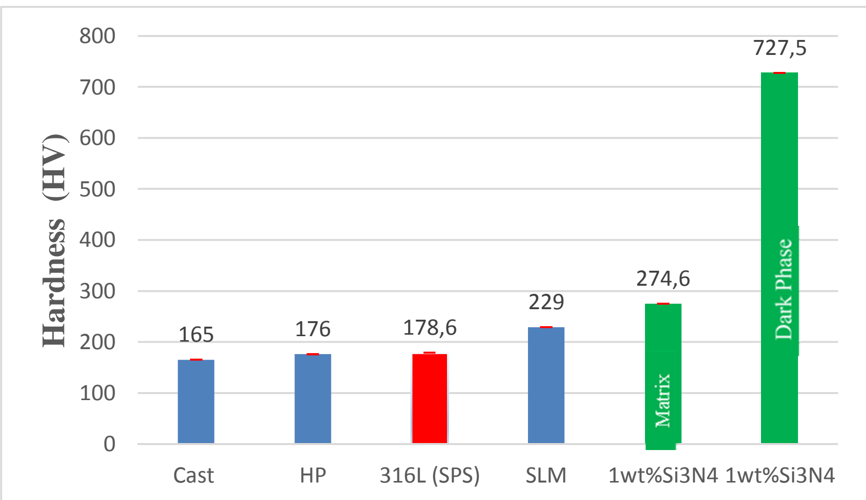

Fig. 4. *Micro-hardness comparison of various materials.*

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The 316L/1wt% Si_3N_4 showed even higher micro-hardness values (Fig. 4). The two present phases (matrix and ceramic) have different hardness values as it was clear from the hardness prints (Fig. 3B). The matrix (Fig. 3B "1") had the hardness of 274.6 HV (66.42% higher than the 316L hardness produced by casting) and the dark phase (Fig. 3B"2") has a hardness of 727.5 HV. The presented higher hardness values in this case are due to the α-Si_3N_4 addition.

The Fig. 5 shows comparison of the friction coefficient curves of tribological measurements lasting 12 hours. A perturbation in the friction coefficient has been observed in both cases. After analyzing the curves, it was noticed that the perturbation started by a drop in the friction coefficient followed immediately by an increase of mean value. A mean friction coefficient of 0.962 (Fig. 5A) has been recorded in the case of the reference sample. The 316L/1wt% Si_3N_4 showed a lower friction coefficient due to its higher hardness, the recorded mean value is 0.803 (Fig. 5B). The perturbation (down and up striations) in the friction coefficient that was observed in both cases was related to the debris formation. The evolution of a metallic debris (in form of SS powder) on the sample surfaces is presented in Fig. 6.

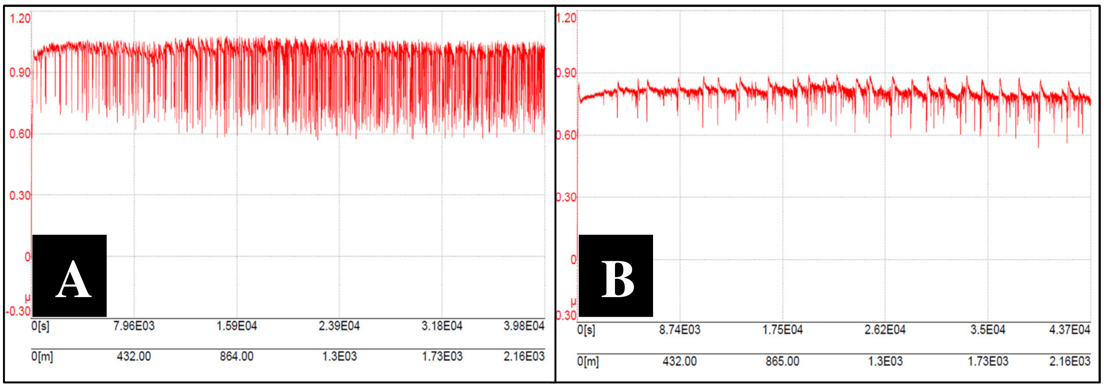

Fig. 5. *Friction coefficient curves after 12hours measurement (2161m. A) 316L reference sample, B) 316L/ 1wt % Si_3N_4*

The decrease in the friction coefficient (Fig. 5 A, B) is taking place when the removed steel grains are stuck under the Si_3N_4 ball and acting as a temporary lubricant before being ejected by the centrifugal force (Fig. 6). This removal of the steel grains from the path of Si_3N_4 balls cause an increase in the coefficient of friction.

The damaged surface on the reference sample after the tribology measurement up to 2161m is shown in Fig. 6. The higher magnification of the damaged area (Fig. 7B) showed the presence of Si_3N_4 originating from the damaged ball (Fig. 8A) embedded in the sample surface and forming a tribolayer.

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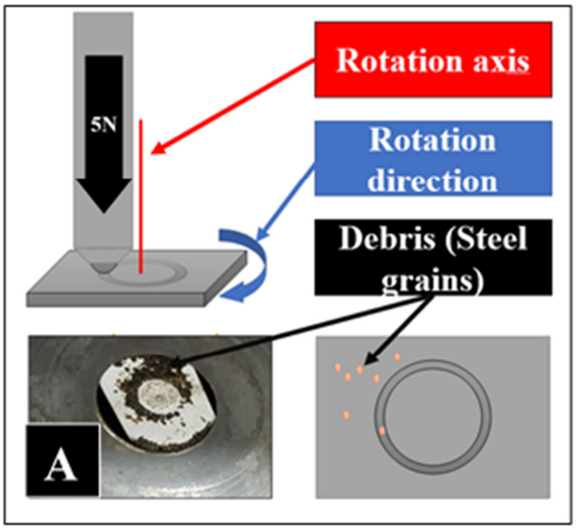

Fig. 6. *Schematic representation of the debris formation*

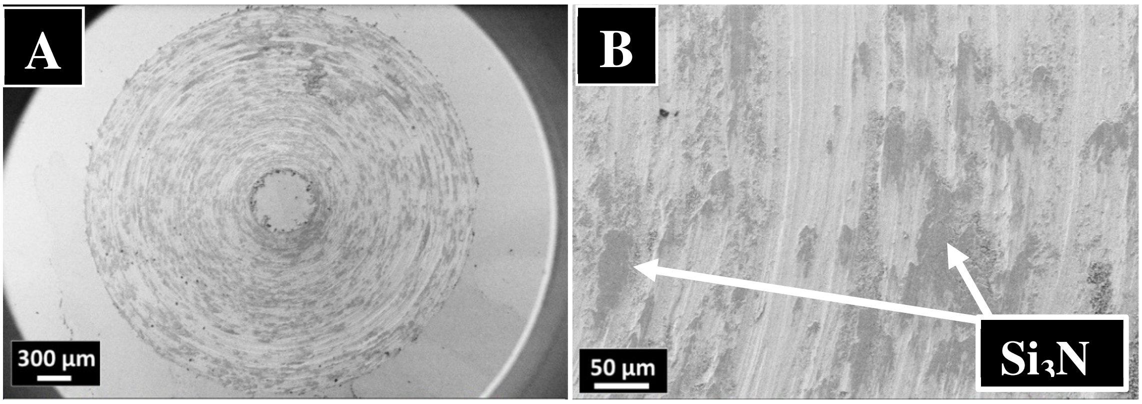

Fig. 7. *SEM image of the 316L/1wt% Si_3N_4 surface damage after 12 hours tribology measurement. A) all wear track. B)detail of wear track.*

The investigation of the damaged ball (Fig. 8A) showed the presence of embedded 316L particles on the eroded ball surface (Fig. 8B).

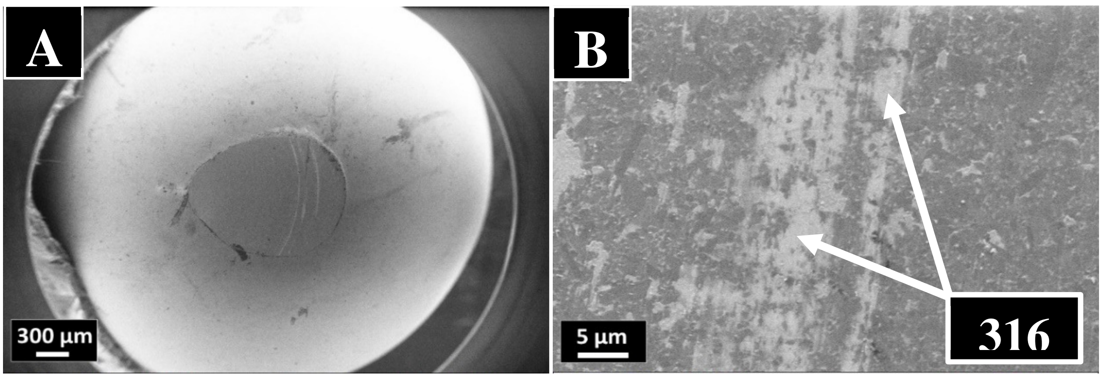

Fig. 8. *SEM image of A) the damaged Si_3N_4 ball, B) Higher magnification of the selected area on (A)*

The Fig. 9A shows the damaged surface on the 316L/1wt % Si_3N_4 sample. The wear track is not symmetrical to rotation axis because this last slightly shifting from its position during the measurement. The higher magnification of the damaged area (Fig. 9B) shows high erosion of the surface together with Si_3N_4 originating from the damaged ball (Fig. 10A) embedded in the sample surface. The investigation of the damaged ball (Fig. 10A) showed the presence of embedded 316L SS particles on the eroded ball surface (Fig. 10B).

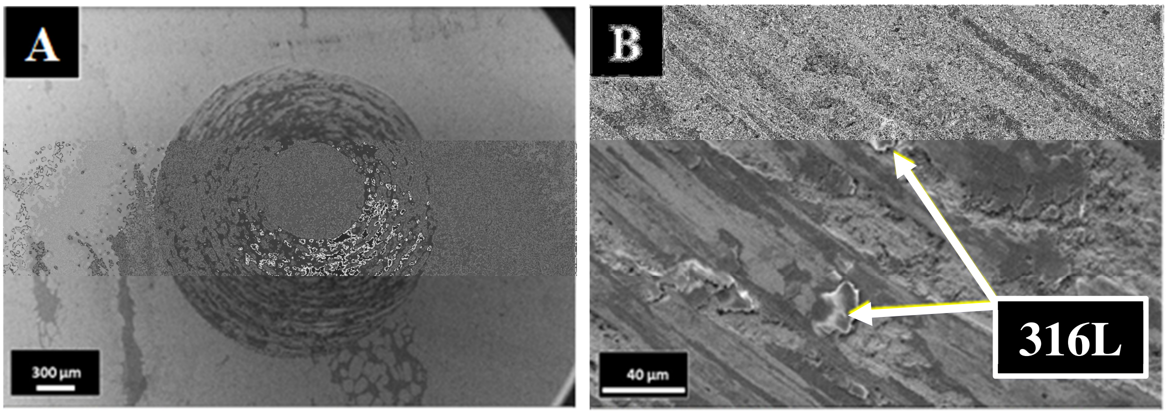

Fig. 9. *SEM images of the 316L/1wt% Si_3N_4 surface damage after 12 hours tribology measurement. A) all wear track, B)detail of wear track.*

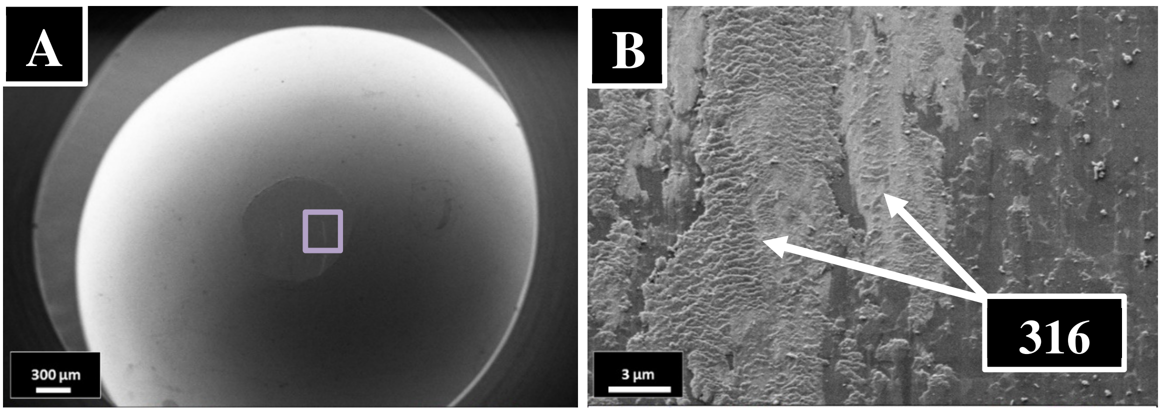

Fig. 10*. SEM images of A) the damaged Si_3N_4 Ball, B) Higher magnification of the selected area on (A)*

4. Conclusion

In this paper the submicron sized α-Si_3N_4 reinforcement has been used to study the effect of ceramic addition on the tribological properties of the commercial 316L stainless steel (SS) prepared by powder metallurgy. Two samples consisting of 316L SS (reference) and 316L with 1wt% Si_3N_4 have been prepared using attrition milling and Spark Plasma Sintering (SPS). The conclusions of this work as the following:

1- The sintered 316L SS reference showed 99.17% relative density

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2- 95.56 % relative density was measured for the sintered 316L/1 wt% Si_3N_4 composite
3- The α-Si_3N_4 addition increased the hardness of the 316L
4- The tribological measurements showed a wear damage on both surfaces of the Si_3N_4 ball and the reference and CDS sample.
5- Perturbation of the friction coefficient has been noticed due to the removed particles/grains during the measurement
6- The decrease in the mean friction coefficient to 0.803 in the case of the 316L/1wt% Si_3N_4 was related to its higher hardness.

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