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# A correlation between nano and micro-hardness properties of TiN nanoparticles strengthened SAF 2205

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**Abstract.** This work studied the correlation between Vickers and nano-hardness of SAF 2205 reinforced with TiN nanoparticles for the convenience of assessing its mechanical properties. Spark plasma sintering was used to fabricate the composites. Micro and nanoindentations were performed to determine micro and nanohardness values of the fabricated composite. Optical microscope was used to assess the microstructure. The microstructure revealed that the TiN dominated the ferrite/ferrite, ferrite/austenite and austenite/austenite grain boundaries. The micro and nanohardness at the TiN dominated grain boundaries were higher compared to the grain hardness. Hardness also increased considerably as the TiN nanoparticles increases. This is also owed to the strengthening effect played by TiN at the grain boundaries by disrupting dislocation motion in the composite. The determined Vickers hardness was plotted as a function of the corresponding nanohardness, a good linear relation was found between Vickers hardness and nanohardness. A linear relationship ( $HV = 215.15 + 15.03H_{\text{nano}}$ ) was established which indicates that there is no difference in behavior for the Vickers hardness and nano-hardness.

## 1. Introduction

The concept of addition of nanoceramics as reinforcement materials in stainless steels has been identified as effective tactics to improve its mechanical properties [1-2]. Various ceramic particles have been explored as potential reinforcement in stainless steels. Of these ceramic reinforcements, titanium nitride (TiN) has emerged as an effective ceramic material that holds high potential for improving the strength of duplex stainless steels. The contribution of grain growth prevention, chemical stability, high hardness and elastic modulus of nitrogen and weight reduction and formability of titanium makes TiN to have excellent properties such as outstanding strength and rigidity, good stability at high temperatures and excellent wear resistance [3-4].

Nanoceramic strengthened stainless steels are developed via powder-metallurgy route which consists of mechanical alloying (MA) with subsequent consolidation via hot isostatic pressing (HIP) or hot extrusion and HP-HT techniques [5]. Recently, MA followed by spark plasma sintering (SPS) was shown to be most suitable approach to fabricate stainless steels-nanoceramics composite. Oke et al. [6] successfully optimize SPS process parameters and fabricated duplex stainless steels reinforced with varied quantities of TiN. The mechanical properties of these developed composites have not been extensively studied.



Amongst available materials testing methods, micro and nano hardness testing are considered to be an efficient means of assessing the mechanical properties of micro or nanoceramics reinforced materials due to the small sampling volume requirement. Hardness has long been regarded as a primary material property and researchers have, over the years, confirmed the importance of high hardness in mitigating wear. However, previous studies have shown that the Vickers hardness value is not equal to the nano-hardness value for the same composite material. A number of studies have been devoted to establish this relation [7-8], but were not precise in giving a fixed correlation. Yang et al [9] studied and established a correlation between Vickers hardness and nano-hardness for the different materials, the authors found a good linear relation was found between Vickers hardness and nano-hardness, and a coefficient was determined to be  $81.0 \pm 10.5$ .

A discrepancy in hardness value attained from micro or nanoindentation test depends on load or indentation depth, different data analysis method could also determine a different hardness value for the same composite materials, which brings difficulty in comparing different experiment results [9]. It is therefore imperative to establish a correlation between Vickers hardness and nano-hardness. In this study, an attempt is made to establish a correlation between Vickers hardness and nano-hardness of spark plasma sintered duplex stainless steel reinforced with TiN nanoparticles.

## 2. Materials and Methods

Commercially available SAF 2205 (C- $\leq 0.03$ , Si- $\leq 1.0$ , Mn- $\leq 2.0$ , P- $\leq 0.03$ , S- $\leq 0.015$ , Cr-22, Ni-5, Mo-3.2, N-0.18, Fe-bal wt.%, average particle size 22  $\mu\text{m}$ , 96 % pure, supplied by Sandvik Osprey Ltd, UK) and TiN (C-0.03, Si- $< 0.003$ , Ni-5, N- $< 21.91$ , Ti-77.83, Fe- $< 0.001$  wt. %, average particle size of 20 nm, 97 % purity, supplied by Nanostructured & Amorphous Materials, Inc. USA) were utilized as the starting powders.

The stainless steel with varying amount (0-8 wt%) of TiN powders were mixed using a turbula mixer (T2F) for 6 hours at 72 rpm to ensure homogeneity. The mixed powders were placed in a graphite die with the inner diameter of 30 mm. An automated spark plasma sintering machine (model HHPD-25, FCT GmbH Germany) was used to consolidate the mixed powders. The composites were sintered in vacuum with pressure of 50MPa, sintering temperature of 1150  $^{\circ}\text{C}$ , heating rate of 100 $^{\circ}\text{C}/\text{min}$  and sintering holding time of 15 min. These sintering parameters were selected based on Oke *et al.* [10]. The microstructural analysis of the samples was carried out using high-resolution optical microscope

Nanoindentation tests were performed using an ultra nanoindenter (UNHT) mechanical testing instrument with a Berkovich diamond indenter equipped with a three-sided pyramid. The testing points of each constituent phase (grains and grain boundaries) could be precisely located by Scanning Probe Microscope (SPM). A maximal load of 1 mN was used and the indenter was held for 10 s so that the time dependent deformation properties can be determined. 10 indents were taken each on the grains and the grain boundary for all the samples. Oliver and Pharr method [11] proposed a common method to determine the hardness of a material from indentation load-displacement data during nanoindentation. Largely, the hardness,  $H$ , is defined as the ratio of the maximum applied load,  $P_{\text{Max}}$ , to the projected area of the indented impression,  $A_p$ , based on the following equations:

$$H = \frac{P_{\text{max}}}{A_p} \quad (1)$$

Vickers hardness tests were carried out using a Vickers microhardness tester (FALCON 500 series) on a polished surface at a maximal load of 100 gf (0.98 N) and dwell time of 15 s at room temperature. For each specimen, 5 measurement tests were conducted and the averaged value was taken as the result.

### 3. Results and discussion

#### 3.1 Microstructural observation

The optical images of sintered Duplex stainless steel with varied amounts of TiN nano particle are shown in Fig. 1. The microstructures consist of austenite, ferrite phases and grain boundaries dominated TiN particles. The TiN particles were noted to increase with TiN reinforcements with a general tendency of the reinforcing TiN phase to locate itself at the vicinity of grain boundaries in the matrix. The dominance of TiN at grain boundaries acts as an effective force in pinning dislocations and avoiding grain growth.

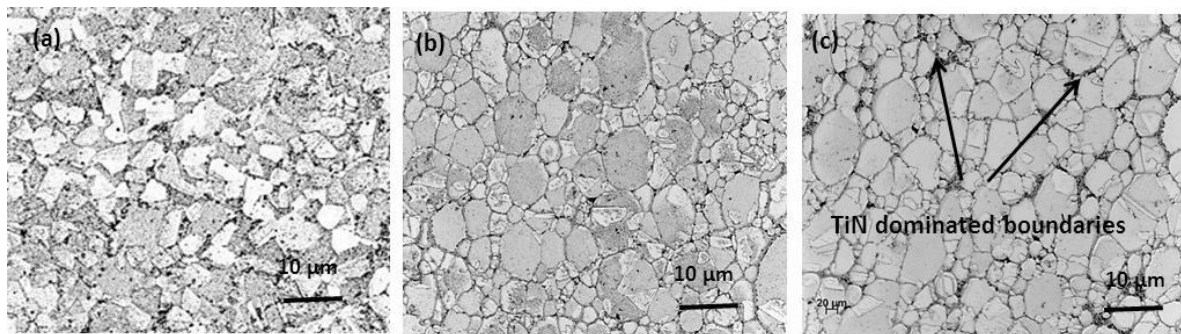


Fig. 1. Optical microstructure showing the effect of TiN on the microstructure: (a) SAF 2205 (b) SAF 2205-4TiN (c) SAF 2205-8TiN

#### 3.2 Grain and grain boundary nanohardness

Nanohardness measurements of the materials were performed with 10 indents made at different locations on the grains (austenite and ferrite) and the grain boundary. The values of hardness (H) of the investigated samples at austenite/ferrite grains and TiN dominated grain boundaries as calculated from the load-displacement curves are presented in Table 1. The grain boundary hardness increased linearly with increasing TiN concentrations from 0-8 %, while the relationship is sinusoidal for grain – the unreinforced SAF 2205 sample have higher hardness than the one with 2% TiN and continued to increase for 4% TiN after which it dropped for 6% TiN while the maximum hardness values was obtained for DSS 2205-8TiN. With increase in TiN content, the role of grain boundaries in blocking dislocation propagation becomes more and more pronounced, resulting in an increased stress concentration at grain boundaries due to dislocation pile-up. Thus, higher applied stresses are needed to propagate dislocations through the material, resulting in a larger hardness [12]. The nature and orientation of the SAF 2205/TiN boundaries can also cause dislocations multiplication, due to this effect there is increase in dislocation density and dislocations tend to hinder the movement of other dislocations, eventually also leading to an increase of hardness [13].

Table 1. Hardness results of SAF 2205/TiN at grains and grain boundaries

Samples	Grain Hardness (GPa)	Grain Boundary Hardness (GPa)
SAF 2205	$4.86 \pm 0.8$	$5.84 \pm 2.0$
SAF 2205-2TiN	$3.72 \pm 1.2$	$12.94 \pm 1.5$
SAF 2205-4TiN	$5.59 \pm 1.5$	$18.71 \pm .08$
SAF 2205-6TiN	$4.91 \pm 1.0$	$22.21 \pm 1.0$
SAF 2205-8TiN	$4.651 \pm 0.5$	$5.2583 \pm 2.0$

### 3.3 Micro-Hardness Properties in Relation to Nanohardness

Fig. 2 shows the results of average micro and nano hardness values obtained from different TiN reinforced SAF 2205 matrix composites. It is observed that the addition of nano particles of TiN to the steel matrix led to significant increase in the micro and nanohardness of composites. In both the cases all composites samples displayed improved hardness compared to that of the unreinforced stainless steel. The lowest micro and nano hardness values were measured as 293.13 VHN and 4.86 GPa respectively for SAF 2205, and the highest hardness values were measured as 476.18 VHN and 16.17 GPa for steel matrix composite with 8% reinforced TiN. Both micro and nanohardness tests displayed similar trends in the increment of hardness values of composites with reference to the unreinforced steel. The increase in hardness values in both cases can be attributed to the high hardness, good dispersion and strengthening effect of TiN nano particles in the steel. It has been reported that good dispersion of reinforcements aids the improvement the interfacial bonding between steel matrix and reinforcements [5, 11]. It is also important to state that the reinforcements could sufficiently resist plastic deformation during indentation.

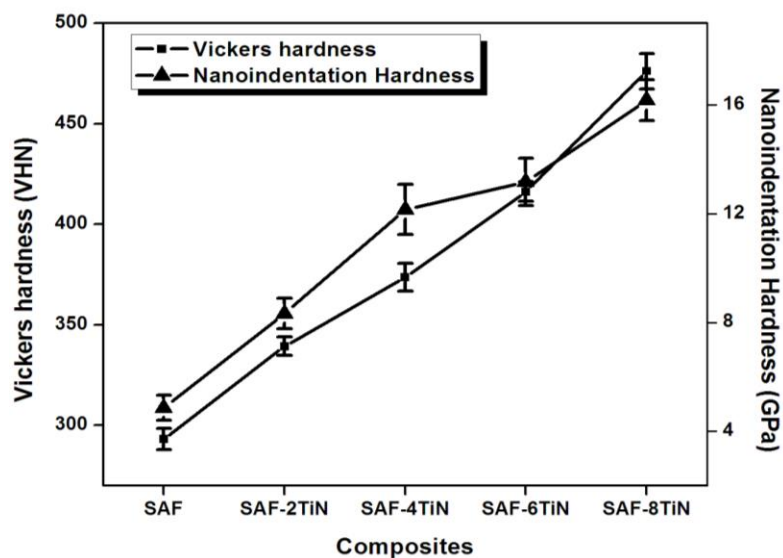


Fig. 2: Micro and nanohardness plots of SAF 2205 and its composites

### 3.4. A correlation between micro-hardness and nano-hardness

The plot of the measured average Vickers hardness as a function of corresponding nano-hardness for the stainless steel composite is presented in Fig. 3. A significant linear relationship is observed to exist between Vickers hardness and nano-hardness. Therefore, line regression can be used to model the linear relationship between Vickers hardness and nano-hardness. The value of the correlation coefficient obtained indicates that the linear relationship is reliable enough to be used to model the relationship of Vickers hardness and nano-hardness for SAF 2205/TiN composites. The deviation in linear fit as noted in Fig. 3 is possibly from the influence of pile-up or sink-in effect occurred during the hardness measurement [9]. The established correlation between Vickers hardness and nano-hardness ( $HV = 215.15 + 15.03H_{\text{nano}}$ ) indicates that there is no difference in behavior for the Vickers hardness and nano-hardness, both of which were obtained from an indentation on materials with an indenter. Yang et al. [9] has established the correlation between Vickers and nanohardness, the authors reported a linear relationship and no significant difference in Vickers and nanohardness. They concluded that the only difference between Vickers hardness and nano-hardness is the definition.

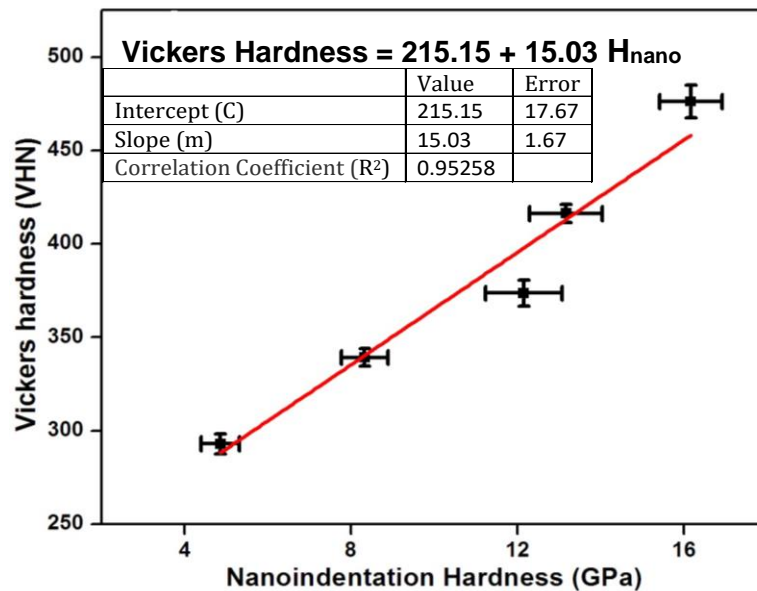


Fig. 3. The Vickers hardness plot as a function of corresponding nano-hardness with increase in TiN nanoparticles.

#### 4. Conclusion

This work studied the correlation between Vickers and nano-hardness of SAF 2205 reinforced with TiN nano particles for the convenience of assessing its mechanical properties. The microstructure revealed the dominance of the TiN nanoparticles at the grain boundaries. Both Vickers and nano hardness exhibited similar trends and were noted to increase with TiN content. There was a good demonstration for the validation of the correlation between Vickers hardness and nano-hardness for the stainless steel composites. Nano-particles and grain boundary strengthening were the dominant strengthening mechanism.

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