

Nanoindentation Studies and Analysis of Mechanical Properties of Ti-4.5wt%Al Based Super Alloys with Fe Additive

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Publication Date: 2026/10/13

Abstract

In this research, nanoindentation tests were carried out on Ti-4.5wt%Al with iron additive at varying weight percentages of 2, 4, 6 and 8 produced using Spark Plasma Sintering technique. The indentation tests were performed on the samples using 300nN and 500Nn indenter loads. Nano-mechanical properties such as modulus (EIT), elastic modulus (E), Vickers hardness (HVIT), creep (CIT), stiffness (S), plastic work (W_p) and elastic work (W_e) using Oliver-Pharr analysis. It was noted that at indentation load of 300nN, maximum displacement value of 2097.5nm was obtained on Ti-4.5wt%Al+4wt%Fe alloy while the least value of 444.43nm was obtained on Ti-4.5wt%+6wt%Fe alloy. It was observed that alloy with 4wt% of iron gave the highest value of both indentation modulus and elastic modulus at both indentation loads. That least creep values of 3.7% and 4.54% were obtained from alloy with 4wt% iron content for both indentation loads of 300nN and 500nN respectively. The displacement (dh) increased with increase in indentation load (dp). Comparatively, Ti-4.5wt%+6wt%Fe has the least displacement-load value, the decrease in the displacement (h) observed on Ti-4.5wt%+6wt%Fe was due to the presence of hard BCC TiFe phase which retards dislocation. Ti-4.5wt%Al+4wt%Fe comparatively has the least combined properties whereas Ti-4.5wt%Al+6wt%Fe has the best combined properties for engineering application.

Keywords: Nanoindentation, Alloy, Elastic Modulus and Displacement-Load.

I. INTRODUCTION

Titanium and its alloys applications in many industries due to high strength to weight ratio and corrosion resistance in many aggressive media are of high demand. They are mainly used in structures in which rather high cost of titanium alloys is compensated by weight reduction of structures and increase in their operational maintenance. The design, development and investigation of the properties of these titanium alloys prior to their usage are of paramount important. Lapin [1], titanium alloys can be in the form of single-phase (TiAl) or two-phase (TiAl) + (Ti₃Al) alloys. The addition of third alloying elements such as Nb or Ta, which gives rise to a single phase alloy, promotes strengthening and enhances oxidation resistance [2,3]. Improvement in ductility can be achieved by the addition of V, Cr, and Mn as well as oxidation resistance by the addition of Niobium and Tantalum or combined properties [3]. Boron and Magnesium can improve hot workability, while Tungsten and Carbon improve creep resistance and oxygen

reduction improves room-temperature tensile elongation [2]. Anioke et.al,[4] reported that increase in the iron content of the Ti-4.5wt%Al alloy, the equiaxed duplex structure ($\alpha + \beta$) Ti phase dominates which led to disappearance of the α_2 -Ti₃Al and formation of an intermetallic phase FeAl₂ which replaced α_2 -Ti₃Al at the grain boundaries. Nevertheless, it became more sophisticated to control the mechanical characteristics of bulk materials or thin films using instrumented indentation at the nanoscale which has been extensively used for a long time,[5]. In load-managed experiments, P-h curves that display exciting local fluctuations or discontinuities are a function of energy-freeing or energy-soaking processes that occur below the indenter tip. [6]. Many engineering materials have been shown to exhibit yield factor at a P-h curve discontinuity that is occasionally referred to as a "pop-in" occurrence,[7]. Conventionally, the weight and intensity of penetration can be measured immediately during loading and unloading of the indenter,[8] which is used to determine the predicted site of contact for the purpose of estimating the mechanical properties of an

engineering material at nanoscale. Using nanoindentation technique, [9], reported that the hardness and elastic recovery of Ti-xFe-yTa ($x = 8, 9, 10$ wt% and $y = 0, 5, 8, 9, 10$ wt%) alloys are within the range of 3.38 to 5.73 GPa and 0.241-0.298, respectively. [10], reported that Mo content material affects the Ti-Mo alloys' segment structure and elastic modulus, that Ti-3.2Mo and Ti-8Mo alloys have low elastic modulus and combination of α and β phases whereas as Ti-4.5Mo, Ti-6Mo, and Ti-7Mo exhibited excessive elastic moduli. [11], reported that Ti-TiB material exhibits higher hardness and elastic values than Ti because of the hardening and stiffening effects of the TiB reinforcements. Hardness and elastic modulus reduced as the nanoindentation load was increased [11,12, 13]. Ti-35Nb-5.7Ta-7.2Zr (TNTZ) was found to have an elastic modulus that is half that of titanium [14]. Values correction length (h_c) growth tend to decrease with material hardness and are highly sensitive to testers' overall performance [12]. In load-managed experiments, P-h curves that display exciting local fluctuations or

discontinuities are a function of energy-freeing or energy-soaking processes that occur below the indenter tip. [6].

Therefore, nanoindentation can be used to study the properties of small volumes of materials, since there are very few literatures on using nanoindentation technique for mechanical characterization of Ti alloys for biomedical application, this work utilizes this method to determine the mechanical properties of the designed Ti-4.5wt%Al+xFe alloys in order to assess the potential of the alloys for engineering and medical applications.

II. MATERIALS AND METHODS

➤ Materials

The raw materials used for this work are Titanium powder (M.W; 48g/mol., Purity;99.5%, Size;45 μ m), Aluminium powder (M.W;27g/mol., Purity; 99.4%, Size; 45 μ m and Iron powder (M.W; 55.86g/mol., Purity; 99.8%, Size; 45 μ m).

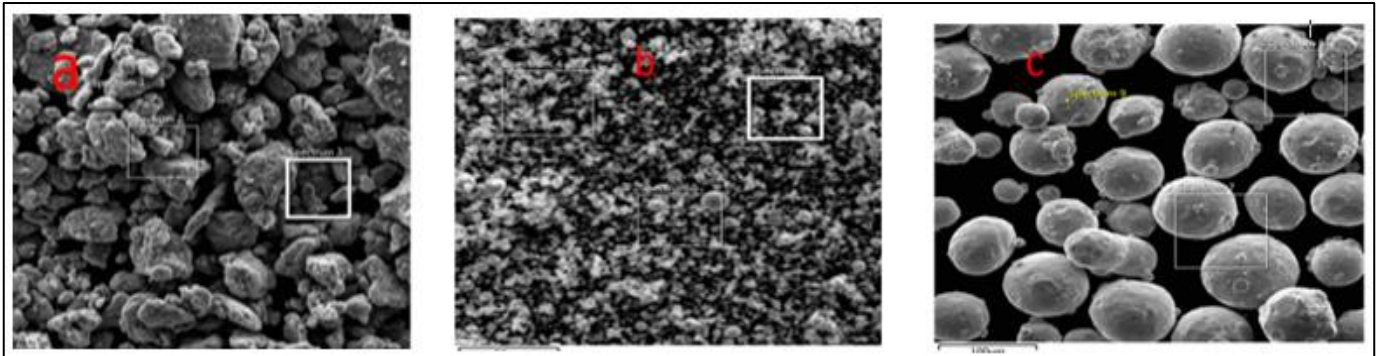


Fig 1 Morphologies of the Elemental Materials (a) Titanium Powder (b) Aluminum Powder (c) Iron Powder

➤ Method

Table 1 Alloy Designs and Process Parameter

S/N	Base alloy wt% (Ti-4.5wt% Al)	Iron (wt%)	Heating rate $^{\circ}\text{C}/\text{Min}$	Sintering pressure MPa	Sintering Temp. ($^{\circ}\text{C}$)	Sintering Time (min.)
1	100 (control)	-	100	30	1000	10
2	98	2	100	30	1000	10
3	96	4	100	30	1000	10
4	94	6	100	30	1000	10
5	92	8	100	30	1000	10

• Alloy Production

The mixing of the elemental materials powder required for the designed alloys as shown in Table 2.1 was done using a mechanical shaker mixer at speed of maximum speed of 500 rpm. The sintering of the mixture was done using spark plasma sintering apparatus (version SPS 10-3) made by Thermal Technologies LLC, the mixed materials were placed between graphite dies and punches while the desired temperature and pressure were applied as indicated in Table 2.1. Dies, punches, and spacers were selected and arranged accordingly. A vacuum of 6 Torr was maintained throughout the experiment. Sample cutting was done using Robofill 240 automatic cutting machine, this was done to avoid over heating of the samples during cutting, the cutting was carried out under water.

• Nanoindentation

Nanoindentation tests were carried out using Anton Parr Ultra-Nanoindenter. The tests were carried out under standard procedures of ISO 14577, test running under constant load to attain a possible maximum depth. Indenting the sample surface mechanically to nm-scale depths while monitoring stress and depth achieves this (indentation). The load and displacement of the indenter were measured as the object was being indented. In order to determine the anticipated area of contact for the purposes of calculating the nano-mechanical properties, load and depth of penetration are typically measured during the loading and unloading of the indenter. The material's nano-properties, including hardness, elastic modulus, creep behaviour, depth-profiling, wear resistance, and fracture toughness, are inferred from these

measurements. The experiment was done using machine defined profile at two different loads of 300nN and 500nN to make the indents at a loading rate of 900nN/min and 1500nN/min respectively for a holding period of 20s. The following properties were evaluated: modulus (EIT), elastic modulus (E), Vickers hardness (HVIT), creep (CIT), stiffness (S), plastic work (W_p) and elastic work (W_e) using Oliver-Pharr analysis.

✓ *Indentation Hardness (HIT)*

The ratio of the applied maximum load F_{MAX} to the resulting contact projected area A_p , or indentation hardness HIT, was calculated [118].

✓ *Indentation Modulus (EIT)*

Although the relationship between applied stress and displacements is no longer linear, it measures the elastic response of a material when subjected to a concentrated load in a single point. As a result, the indentation modulus represents a good approximation of the Young's modulus. (ISO 14577-1 Annex A.5; Metallic Materials) The indentation modulus EIT is a function of a number of variables and boundary conditions.

$$EIT = \frac{1 - V_s^2}{\frac{2\sqrt{A_p}}{S\sqrt{\pi}} - \frac{1 - V_i^2}{E_i}}$$

Where v_s is the sample's Poisson ratio, V_i and E_i are the indenter material's Poisson ratio and Young's modulus, respectively, S is the contact stiffness, or the increment ratio between unloading and related displacement at the maximum depth of indentation, and A_p is the projected contact area.

✓ *Indentation Creep (CIT)*

The fluctuation in indentation depth, as determined by a constant test force applied over a predetermined period of time, is caused by the material's creep. From the following equation, it is calculated as the relative change of the indentation depth (ISO 14577-1 Annex A.6; Metallic Materials)

$$CIT = \frac{h_2 - h_1}{h_1} \times \frac{100}{1}$$

Where h_1 is the indentation depth when the test force is reached at time t_1 and is kept constant, and h_2 is the indentation depth when the test force is held constant at time t_2 , where $h_2 = h_{max}$

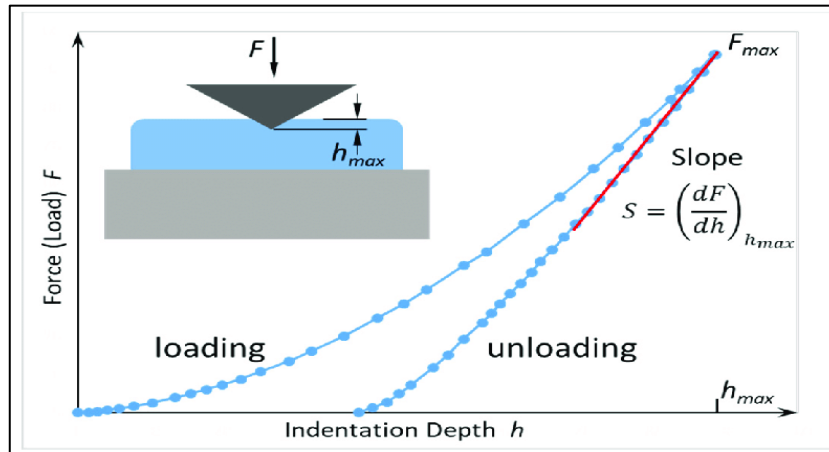


Fig 2 Load-Displacement Curve Schematically for the Nanoindentation test [8].

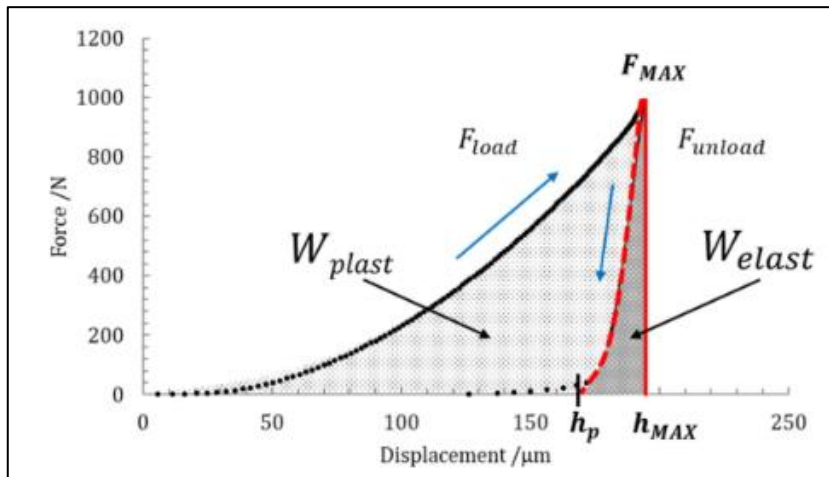


Fig 3 Experimental Regions for the Elastic and Plastic Components of Indentation Work under Loading and Unloading Indentation Curves [15,16]

III. NANO-MECHANICAL ANALYSIS

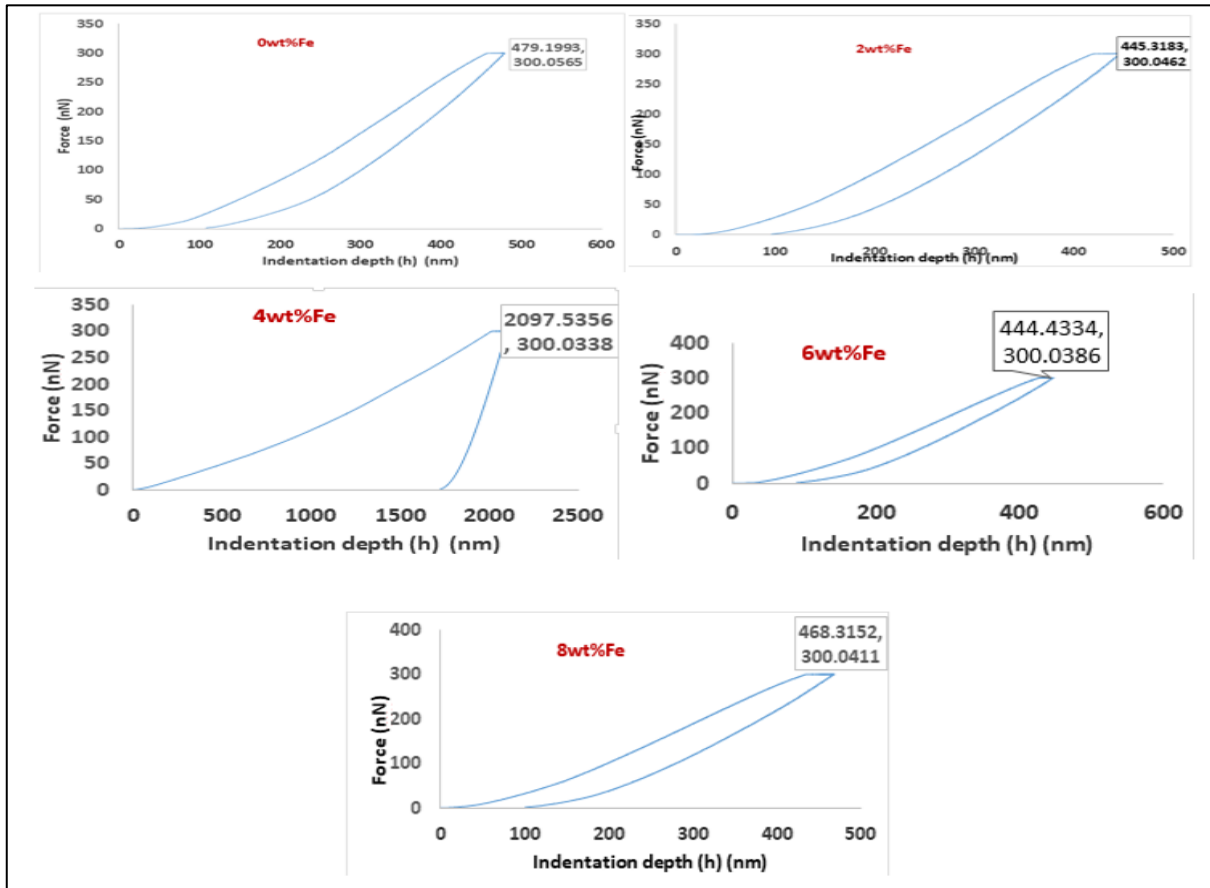


Fig 4 Load-Displacement Curve of Ti-4.5wt%Al+ (2,4,6 & 8wt%Fe) at 300nN Maximum Load

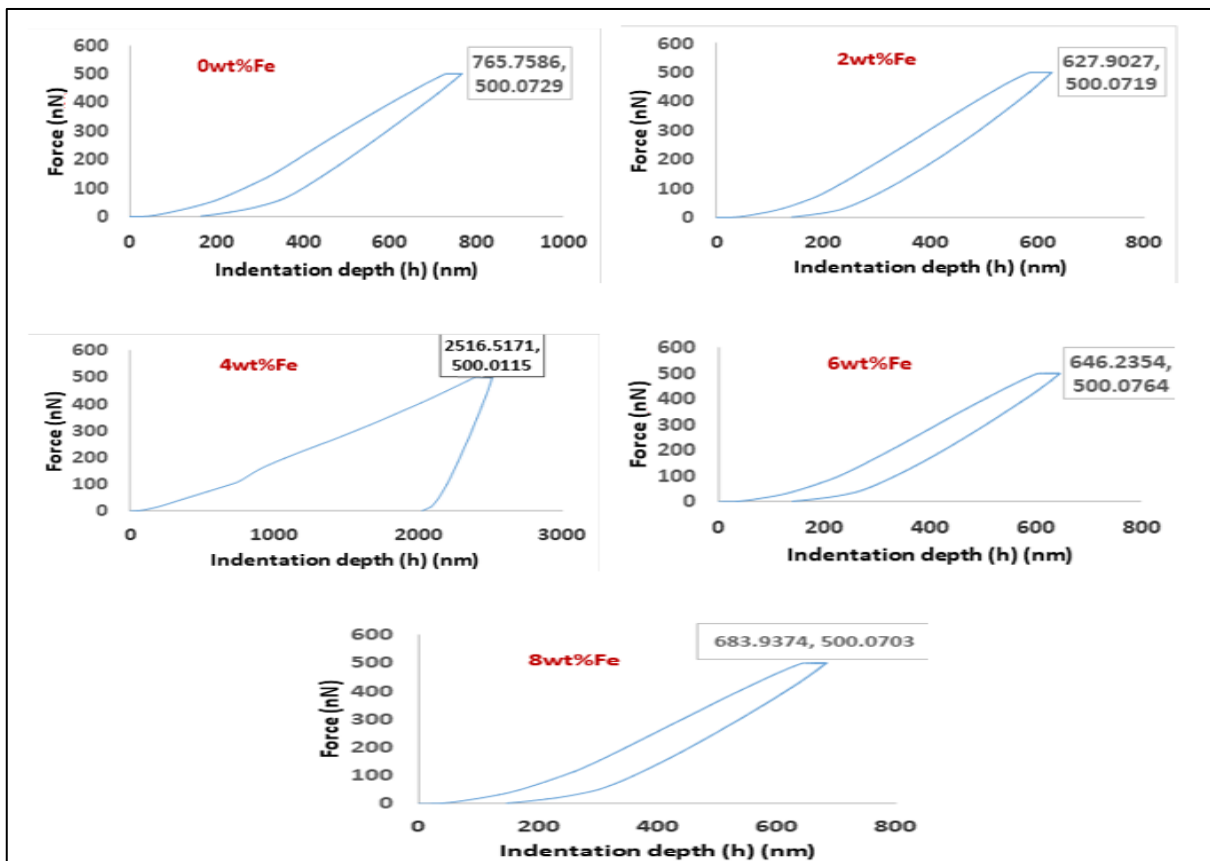


Fig 4 Load-Displacement Curve of Ti-4.5wt%Al+ (2,4,6 & 8wt%Fe) at 500nN Maximum Load

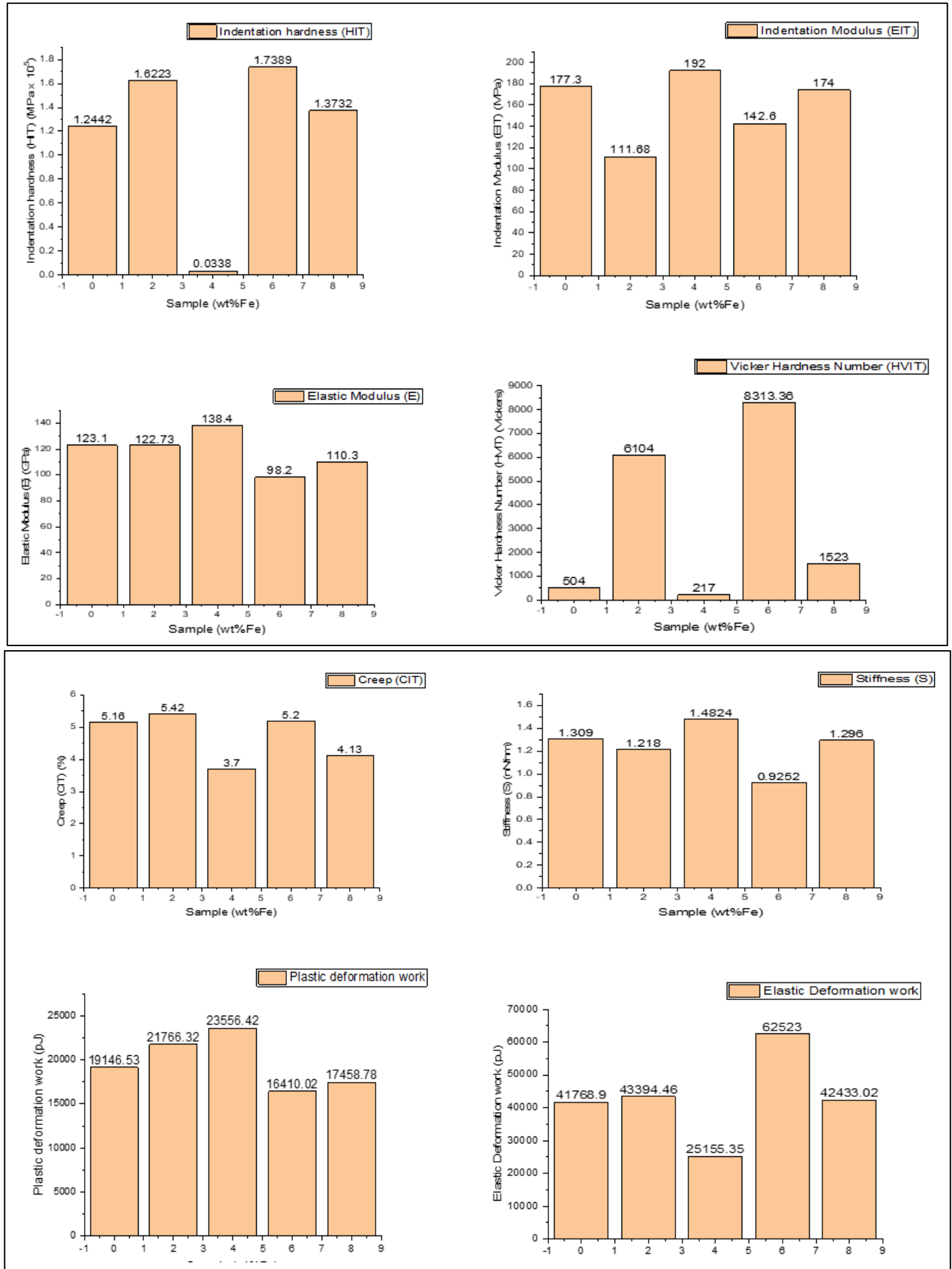


Fig 5 Nano-Mechanical Properties of the Alloys at 300nN

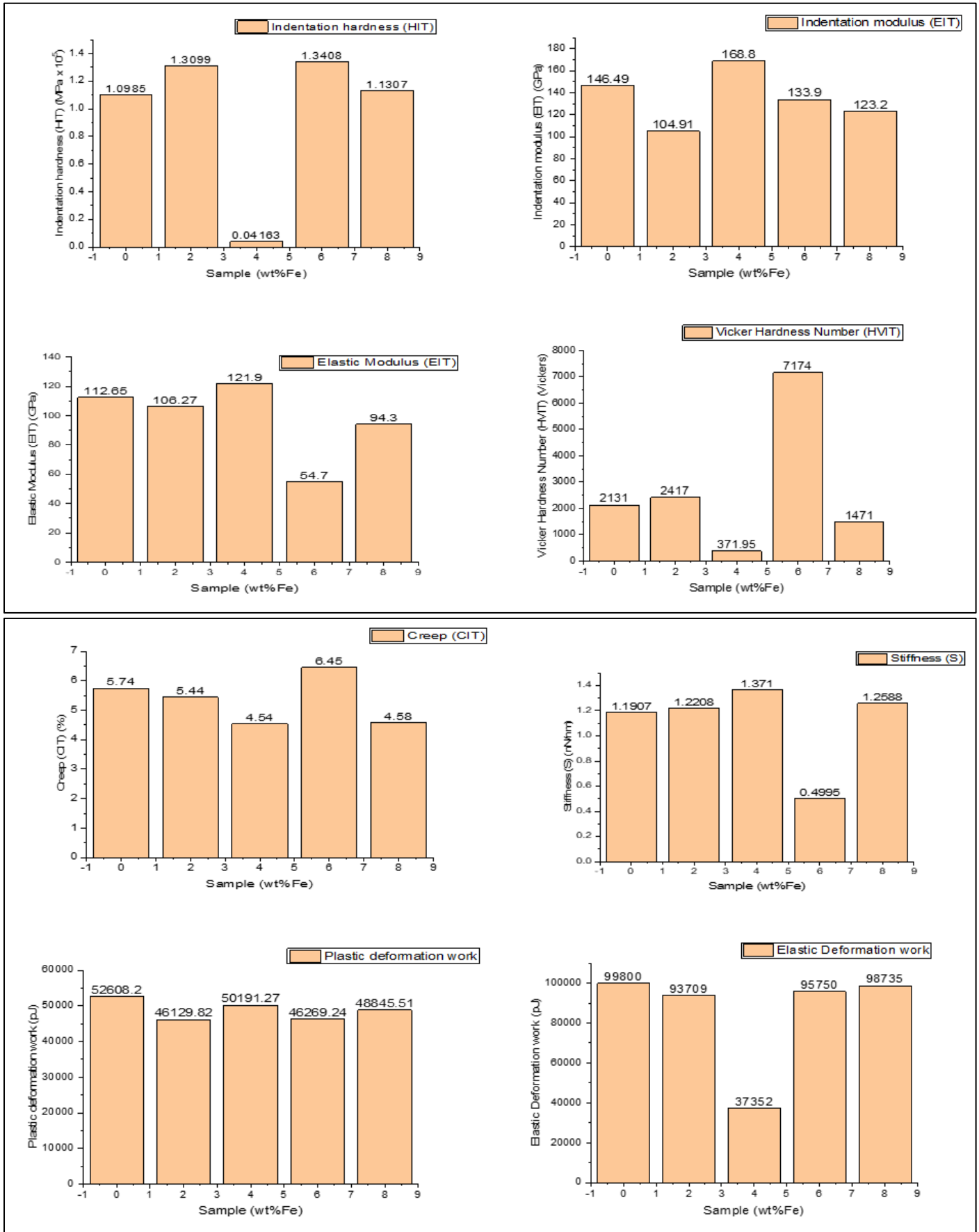


Fig 6 Nano-Mechanical Properties of the Alloys at 500nN

➤ Load-Displacement (dp/dh)

The nanoindentation load-displacement curves of the master alloy and alloys with iron additive under indentation load of 300nN and 500nN are presented in

figures 3 and 4 respectively. The graphs typically show the loading and un-loading behavior showing smooth curves without pop-in effects at indentation loads. It was observed that at indentation load of 300nN, maximum displacement

value of 2097.5nm was obtained on Ti-4.5wt%Al+4wt%Fe alloy while the least value of 444.43nm was obtained on Ti-4.5wt%+6wt%Fe alloy. This increase observed on the Ti-4.5wt%+4wt%Fe alloy can be attributed to the delaminating phenomenon of the particulars of the alloy which was caused by the present of $Al_3Ti_{0.75}Fe_{0.25}$ in the alloy which allowed the easy penetration of the indenter into the materials as reported by Anioke et. al. However the decrease in the displacement (h) observed on Ti-4.5wt%+6wt%Fe was due to the presence of hard BCC TiFe phase which retards dislocation as reported by Anioke et. al.

At indentation load of 500nN it was observed that Ti-4.5wt%+4wt%Fe alloy gave maximum displacement of 2516.5nm while Ti-4.5wt%+2wt%Fe gave the least displacement of 627.9nm, these behavior can be attributed to the presence of phases as explained above. It was also observed that displacement increased with increase in indentation load.

➤ *Indentation hardness (HIT) (MPa) and Vickers hardness number (HVIT) (Vickers)*

Figures 5 and 6 show the indentation hardness and Vickers hardness number of the alloys under 300Nn and 500Nn load respectively. It was observed that high indentation hardness and Vickers hardness were obtained from Ti-4.5wt%+6wt%Fe alloy for both indentation loads of 300nN and 500nN. Moreso, it was observed that least indentation hardness and Vickers hardness were obtained from Ti-4.5wt%+4wt%Fe alloy at both indentation load, these behavior can be attributed to the presence of the phases as stated above. It was also observed that the indentation hardness varied with variation in the iron content.

➤ *Indentation Modulus (EIT) (GPa) and Elastic Modulus (E) (GPa)*

Figures 5 and 6 show the results of the indentation modulus and Elastic modulus at both loads of 300nN and 500nN respectively. It was observed that both indentation modulus (EIT) and elastic modulus (E) changed with change in iron content of the alloys. Comparatively, it was observed that both indentation modulus and elastic modulus decreased with increase in the indentation load. However it was observed that alloy with 4wt% of iron gave the highest value of both indentation modulus and elastic modulus at both indentation loads.

➤ *Creep (CIT) (%)*

Figures 5 and 6 show the results of the indentation creep (CIT) of the alloys, it was observed that least creep values were obtained from alloy with 4wt% iron content for both indentation loads of 300nN and 500nN. Whereas the highest creep values were obtained from alloy with 2wt%Fe and 6wt%Fe at indentation loads of 300nN and 500nN respectively. It was observed that the indentation creep varied with variation in iron contents of the alloys. It was noted that the indentation creep increased with increase in the indentation load.

➤ *Stiffness (S)*

Figures 5 and 6 show the results of alloy stiffness (nN/nm) at both indentation loads respectively. It was observed that alloy with 2wt%Fe gave the maximum stiffness at indentation load of 300nN while alloy with 6wt%Fe gave the maximum value at 500nN indentation load. It was also observed that the stiffness decreased with increase in the indentation load, also the stiffness at constant indentation load varied with variation in the iron content.

➤ *Plastic Deformation Work (PJ) and Elastic Deformation Work (PJ)*

Figures 5 and 6 show the result of plastic deformation works and elastic deformation works at indentation load of 300Nn and 500Nn respectively. It was observed that the maximum plastic deformation works were obtained from alloy with 4wt%Fe, can be illustrated with area under the graph of loading and unloading curves from the load-displacement graphs at indentation loads of 300nN and 500nN. Comparatively, areas under the load-displacement graph for this alloy is larger than others. These show that a constant indentation loads of 300nN and 500nN, greater works were done on the alloy compared with other alloys, this phenomenon agrees with the reports of [15,16]. The elastic deformation work done on alloy with 4wt%Fe gave the least values at both indentation loads as shown in figures 3 and 4, this also agrees with report of [15,16].

IV. CONCLUSION

Nanoidentation analysis was used in this study to evaluate the nano-mechanical properties Ti-4.5wt%Al alloy with 2wt%, 4wt%, 6wt% and 8wt% iron additive produced by spark plasma sintering technique.

➤ *The Following Conclusions are Drawn:*

- That the maximum plastic deformation works were obtained from alloy with 4wt%Fe
- That at indentation load of 300nN, maximum displacement value of 2097.5nm was obtained on Ti-4.5wt%Al+4wt%Fe alloy while the least value of 444.43nm was obtained on Ti-4.5wt%+6wt%Fe alloy.
- That displacement (dh) increased with increase in indentation load (hp)
- That high indentation hardness and Vickers hardness were obtained from Ti-4.5wt%+6wt%Fe alloy for both indentation loads of 300nN and 500nN.
- That both indentation modulus and elastic modulus decreased with increase in the indentation load.
- That least creep values of 3.7% and 4.54% were obtained from alloy with 4wt% iron content for both indentation loads of 300nN and 500Nn respectively.
- Stiffness at constant indentation load varied with variation in the iron content.
- Maximum plastic deformation works were obtained from alloy with 4wt%Fe.
- That Ti-4.5wt%Al+4wt%Fe comparatively has the least combined properties whereas Ti-

4.5wt%Al+6wt%Fe has the best combined properties for engineering application.

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