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Examination of Modulus of Elasticity (E), and Strength Criteria of Beta Phase(B) Titanium/Copper (TiCu) Alloy for Intrauterine Gynecological Application

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ABSTRACT

The purpose of this experimental research study was to investigate the modulus of elasticity (E) of beta-phase (β) Titanium Copper alloy (TiCu) specimens for intrauterine contraceptive applications. In this study, we design and produced Beta (β) phase TiCu alloy specimens with varying copper percentages, Ti0.5%Cu, Ti1.0%Cu, Ti2.0%Cu, Ti5.0%Cu, Ti10.0.0%Cu, Ti15.0%Cu, and Ti17.0% Cu, using copper as the experimental control reference biomaterial. The samples were produced using the powder metallurgy technique in an inert environment. Experimental investigations and Minitab Software Design analyses were conducted to examine the combined load variation with strain rate conditions on the specimens. Furthermore, the study examined the correlation between stress and strain rates in the endometrium. The elastic modulus of the Titanium Copper alloy confirmed optimal stress-to-strain values, which are crucial in the design of biomaterials for surgical implants under load conditions. The strain developed in the alloy determined its deformation under different forces and fatigue conditions, considering the periodic loads on the implant material. The results indicated that the Ti17%Cu alloy specimen exhibited an excellent modulus of elasticity value. The research establishes the strength criteria, particularly the acceptable modulus of elasticity (E), and suggests that the Ti17%Cu alloy could be a viable replacement for the existing prototype biomaterial (Cu-T380 IUD) based on its optimal modulus of elasticity. These strength properties are essential for ensuring the reliability and durability of intrauterine biomaterials in the cervix. We reported that this development holds significant promise in enhancing the mechanical performance and compatibility of intrauterine devices, thereby contributing to advancements in women's healthcare.

Keywords: biomaterials, contraceptive, titanium copper alloy, gynaecological, beta phase, specimens, endometrium, Cu T380IUD, intrauterine

1.0 Introduction

Modulus of Elasticity effect on mechanical hardness and work hardening process is considered a dominant feature in biomaterial because of the desired increase in resistance against wear and corrosive effects of body fluids (Udeh *et al.,* 2022; Ezeh *et, al.*,2020). The machining operations like milling and drilling cause undesirable work hardening in Ti and Ti-based alloys, and with this associated machining, there is an increase in hardness. As a result of this undesired manufacturing process, it creates a negative effect on the quality of finished end products. In vivo, research involving implanting Ti alloys in rabbits showed that the hardness and fracture toughness of Ti-5Al-2.5Fe and Ti-6Al-4V-ELI were not affected before and after 11 months of implantation in the host environment due to the investigation that proved the microstructure and modulus of elasticity remaining unchanged and very good for strength load bearing (Sammons,2011; Ezeh *et. al*;2020). Investigative research proved that binary Ti-xTa alloys with Ta content increase up to 0-50 wt.% Ta, the microhardness property initially diminished and then increased, and finally decreased again as the percentage kept increasing. For the ternary alloy, Ti-20Nb-xTa, when there is an increase in Ta content in the range of (0-10 wt.% Ta), the modulus became constant, while the microhardness initially decreased and then increased (Erlin,2013; Ezeamaku *et, al*;2022). These changes occurred due to fluctuations of the very surface deformation layers, as well as ternary Ti with alph+beta phase, having a higher hardness and lower tensile strength than binary Beta phase Titanium alloy (Amir,2015).

Biomaterial research reports established that human tissue is mainly organized of self-assembled polymers (proteins) and ceramics (bone materials), having metal constituents as trace elements (Qizh and George, 2015). These trace metal elements are Cu^{2+}, Zn^{2+}, Ag^{2+} e.t.c, which create contraceptive response resulting in non-motility of the spermatozoa and inability of ova fertilization by the principle of actions of inhibition of both glycolytic oxygen and glucose uptake by the spermatozoa (Udeh *et al.,* 2022). The application of metals and alloys for biomedical contraceptives within the endometrium host environment is a research innovation, not yet been explored fully, a biomedical expert, reported that heavy metal ions possess spermicidal properties, and anchored this research finding on the effects of Cu^{2+}, Zn^{2+}, Fe^{2+}, Pb^{2+}, Cd^{2+}, Mn^{2+}**,** and Co**.** Titanium has a phase transformation from alpha to beta phase at temperatures above 883^{0}C. Below 882.5^{0}c, Titanuim exists as alpha-phase (α) material and the crystal structure is hexagonal close-packed (Hcp), but above 883^{0}c it changes to the body-centred cubic system(bcc) in beta(β) phase, because it possesses high passivity and regenerative properties i.e the ability to repair itself and form protective covering, with dense oxide film(Coating,2003).

2.0 Materials and Methods

2.1 Identification of problems with intrauterine contraceptive biomaterials

The problems of existing Intrauterine implants (CuT380) are expulsion, fragmentation, corrosion, and infection phenomena. Figure 1 depicts the insertion of CuT380 in the endometrium. The occurrence of biodegradation and loss of material integrity is due to corrosion and wear created by the vivio human environment, thereby influencing the mechanical properties of the biomaterial and usually leading to mechanical failure, an index of the modulus of elasticity (E)of the material (Gilbert,1993).

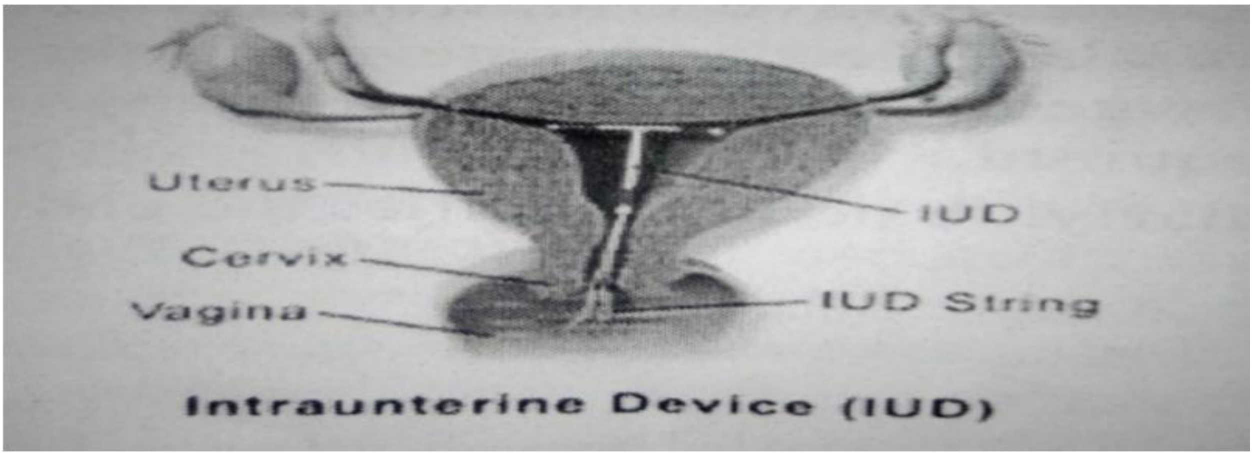


Figure 1: Insertion of IUD T380 in the Endometrium (Kalpana Gupta, 2009)

2.2 Development of Samples (Ti-Cu) By Powder Metallurgy

The development and analysis of the beta phase Ti-Cu alloy (Bcc) specimens were innovated, using the highlighted characterized parameters which influenced the acceptability of the researched alloy, Titanium Copper alloy (Paul *et al*, 1988). Titanium as an element is allotropic, existing in more than one crystalline form, which at room temperature is Hexagonal close-packed (HCP) and of Alpha phase (Amir *et al*, 2015), but when alloyed with a Beta phase stabilizer like copper, at a temperature of 928°c-1005° c to form Titanium Copper alloy, there is a metallurgical phase transformation to Beta phase with Body-centered cubic structure (Bcc) (Udeh, 2021).

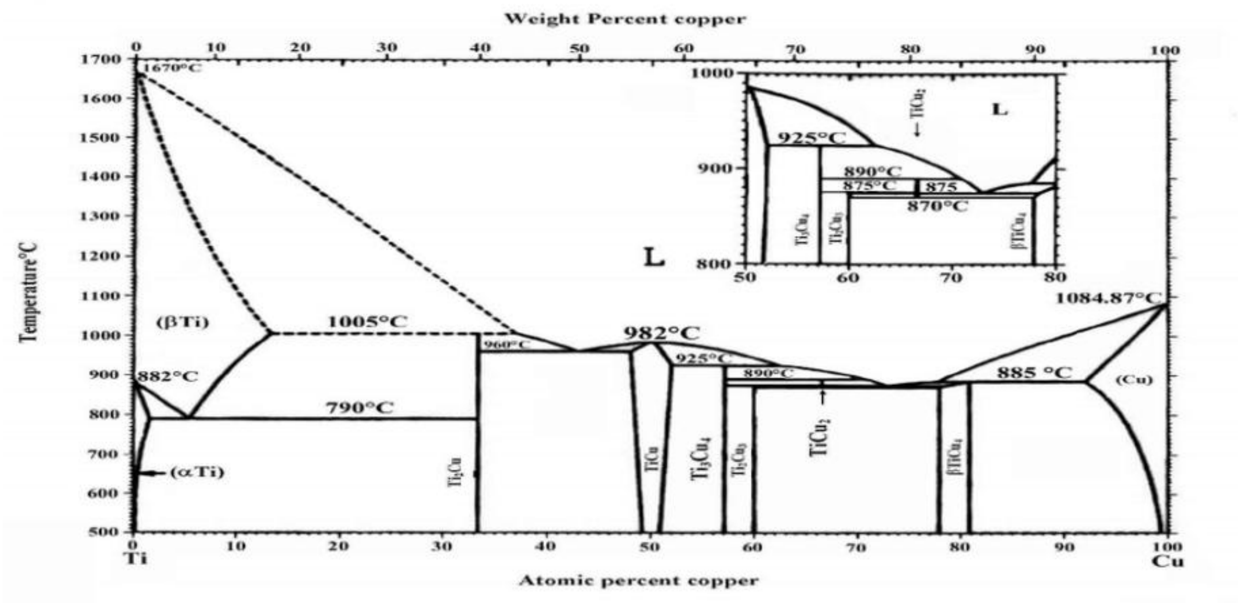


Figure 2: Phase Diagram of Ti-Cu Alloy (courtesy, Good fellow Inc, USA) (Udeh *et al.,* 2022)

The alloy samples were prepared using the Powder metallurgy approach. Commercial pure copper powder named (cpCu) was used for the alloying, and also for the manufacture of control reference biomaterial (100%Cu). In the material design, the production of TixCu alloy specimens (x=0.5%,1.0%,2.0%,5.0%,10.0%,15.0%, and 17.0%) is by powder metallurgy in an inert environment at eutectic maximum solubility of 17.0% copper in beta Titanium phase at 1005^0 C and compaction pressure of 500MPa, as depicted above in the phase diagram of Titanium Copper alloy. The copper element (control reference material) powder is also processed in an inert environment at the temperature of 1005^0 C (Udeh, 2021). The Titanium powder and copper powder were each weighed out differently, and ball-milled differently for 4-7 hrs., and then were pressure compacted up to 500MPa, to develop the specimens (TiCu Alloy), being 30mm in diameter, and under vacuum conditions of 983^0 c -1005^0 c for 135-190 minutes, and allowed t cool in furnace to room temperature of 30^OC. The thermocouple inserted into the bottom punch was used to measure the temperature. Titanium-copper alloy (TiCu) specimens were prepared from Titanium powder and Copper powder at (99.5% purity), with different percentage weight compositions as follows: (99.5% Ti 0.5%Cu), (99%Ti 1.0%Cu), (98.0% Ti 2,0%Cu), (95.0% Ti 5.0%Cu), (90.0% Ti 10.0%Cu), (85.0% Ti 15%Cu), (83% Ti 17%Cu). Specimens of a diameter of 30mm and a thickness of 2.5cm were sliced off from the TiCu specimens for the various tests using dies and punches of graphite.

The manufacturing option of powder metallurgy approach of beta-phase Ti-Cu alloy specimen was very clinically acceptable due to its high degree of affinity with tissues in the endometrium. Amir *et al.* (2015); Ezeh *et al.*2019, collaborated on the results of Titanium Copper alloy fabrication and adopted the powder metallurgy approach of this research.

Figure 3: Developed beta-phase Titanium copper specimens and copper specimens (Udeh *et al.*, 2022)

Table 1: Composition of samples used

Specimen	Composition
1	Ti-0.5%Cu
2	Ti-1.0%Cu
3	Ti-2.0%Cu
4	Ti-5%Cu
5	Ti-10%Cu
6	Ti-15%Cu
7	Ti-17%Cu
8	100%Cu

3.0 Experimental

3.1 XRD Phase and Microstructure

The X-ray diffraction (XRD) analysis and Scanning Electron Microscopy (SEM) Microstructure examination of the Titanium copper alloy specimens were conducted**.** The SEM microstructure analyses of the samples at the various compositions of copper in the Titanium matrix at TixCu (x= 0.5, 1.0, 2.0, 5.0, 10.0, 15.0, and 17.0) were observed. Mechanical Strength Test for Establishment of Modulus of Elasticity(E).

3.2 Microhardness test

The Microhardness test was handled by first polishing the specimens, and the point of indentation mark occurrence on their surfaces was recorded by the indentation machine. The value of the Brinnel Hardness test is read off from the indentation machine, and recorded for each specimen of cylindrical dimension of 30mm and thickness of 25mm.

3.3 Tensile test

The tensile and percentage elongation tests on the specimens were handled with the universal testing machine (Onukwuli *et, al*;2018). The machine has a loading frame, and a controller plotter, with progressive strain rates of 0.010/sec, 0.015/sec, 0.020/sec, 0.025/sec, and 0.030/sec. The loads versus strains on each specimen were recorded.

3.4 Fracture Toughness Test

The fracture toughness test on the specimens progresses with surface polishing to remove any indentation, and then an initial crack or v-groove notch is made on the surface of the specimen. The gradual loading on the machine commenced, and the load at which the test piece fails to resist crack propagation is recorded for each specimen.

4.0 Results and Discussion

The Data analysis adopted an SPSS/ Excel flow sheet for graphical illustrations of the results. The determination of variance and the regression relationship between the Ti Cu alloy test results and copper element are verified using the MINITAB approach to establish and validate the results

4.1 XRD Phase and Microstructure

The XRD and SEM microstructure analyses indicated the formation of Ti_2Cu inter-metallic beta phase, with Bcc structure for all the beta phase Titanium copper alloy specimens.

Scanning electron microscopy (SEM) showed the presence of inter-metallic Titanium copper (Ti2Cu) which provided the interface for mechanical strength and good biocompatibility properties (Hudson, 2012; Sykaras, 2000; Ezeh *et. al*;2019). Figure 4 shows the XRD pattern of Ti 17% Cu and copper element. it indicated new peak identification at 40° and 70° for the Titanium Copper element. The result is following the findings of Qizh and George (2015) that the XRD and SEM microstructure analyses indicated the formation of Ti_2Cu inter-metallic beta phase, with Bcc structure for all the beta phase Titanium copper alloy specimens.

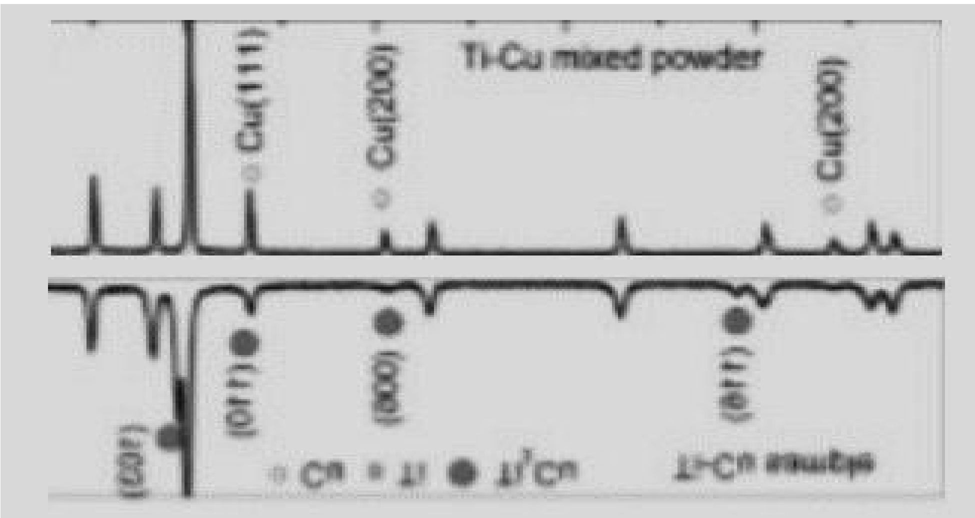
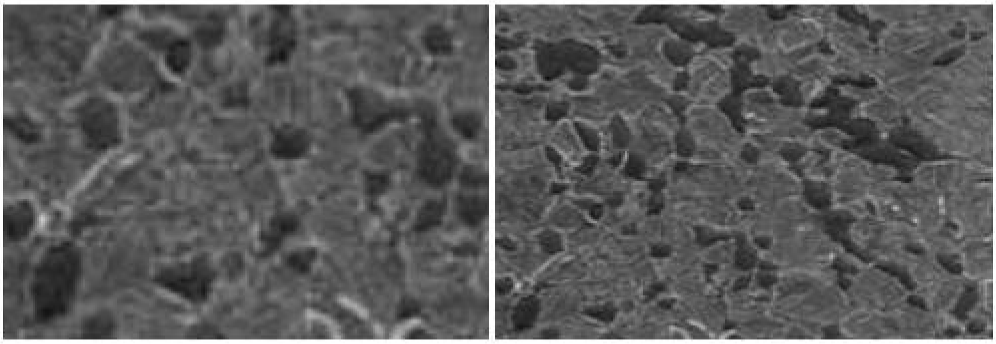
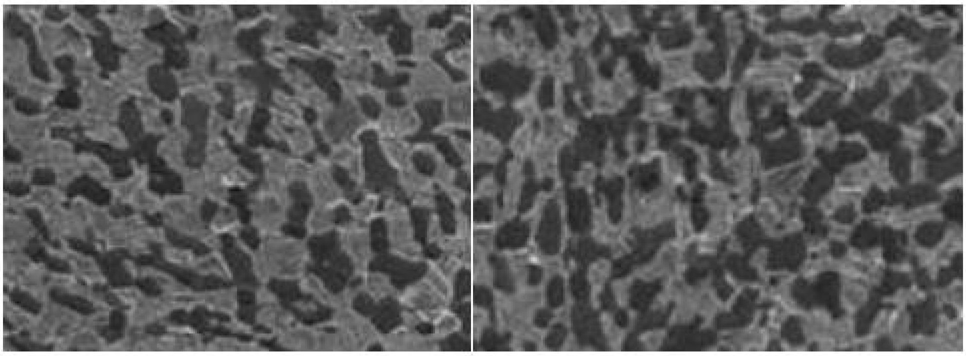


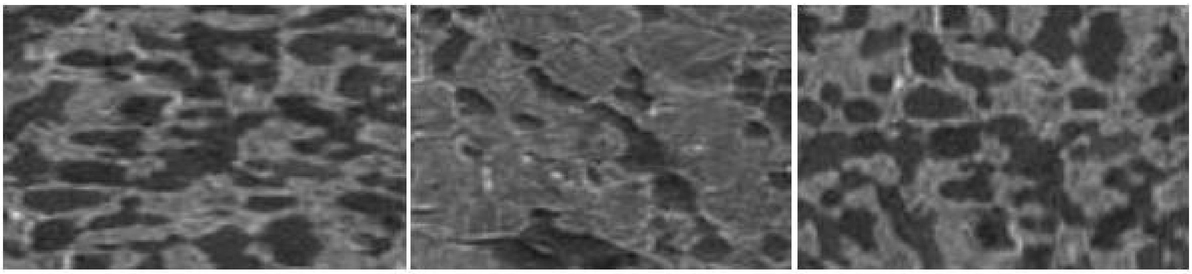
Figure 4: XRD pattern of Ti 17% Cu and copper element



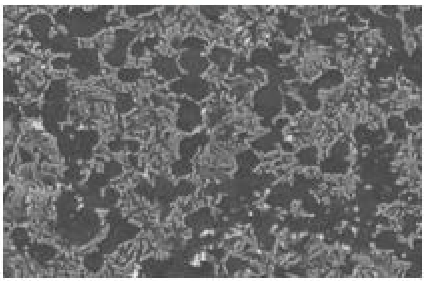
(a)Ti0.5%Cu *(b) Ti1.0%Cu*



(c)Ti2.0%cu *(d) Ti5.0%Cu*



(e)Ti15.0%Cu *(f) Ti10.0%Cu* *(g) Ti17.0%Cu*



(h)100%Cu

Figure 5: SEM Microstructure Examinations

4.2 Tensile strength

The Plots of Tensile stress versus strain for the various specimens (Ti 0.5%Cu, Ti 1.0%Cu, Ti2.0% Cu, Ti 5.0%Cu, Ti 10.0%Cu, Ti 15.0%Cu, Ti 17%Cu and 100%Cu) are shown below.

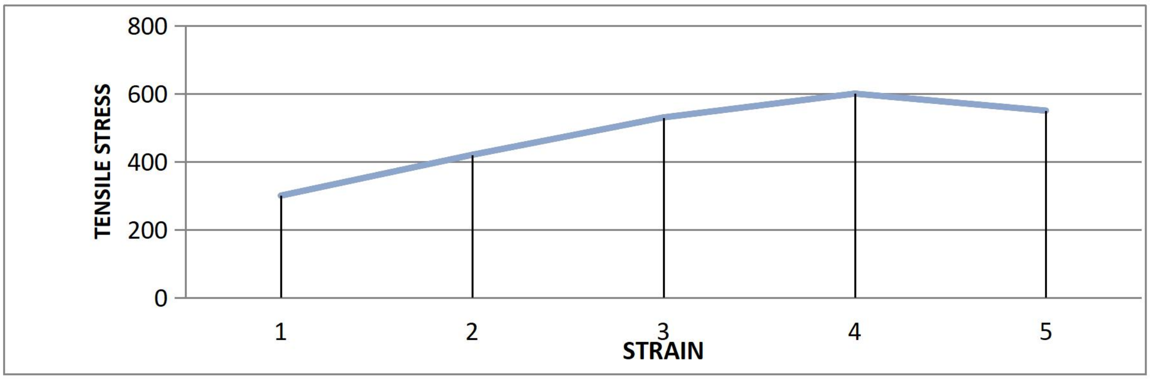


Figure 6: Tensile test (Ti0.5%Cu)

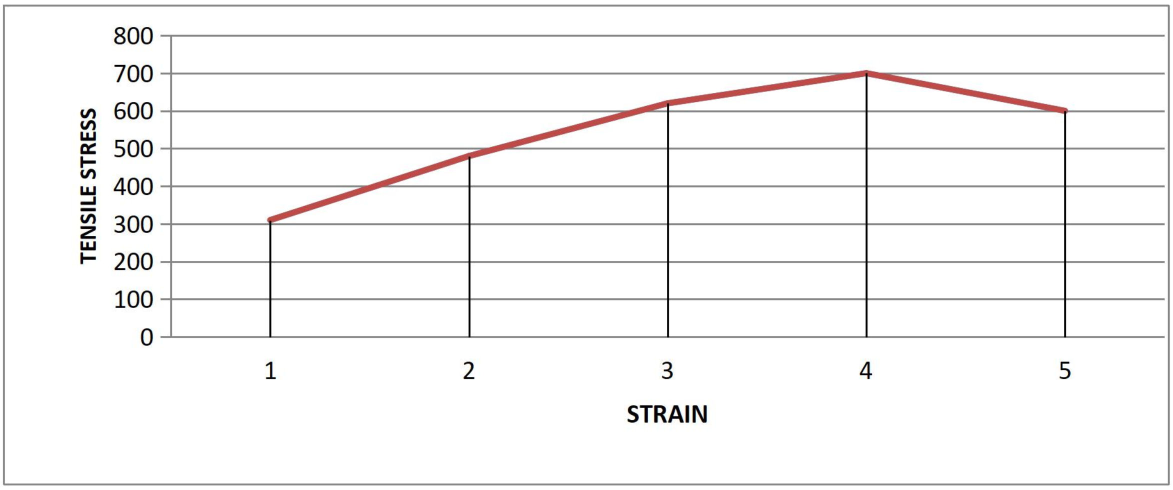


Figure 7: Tensile test (1.0%Cu)

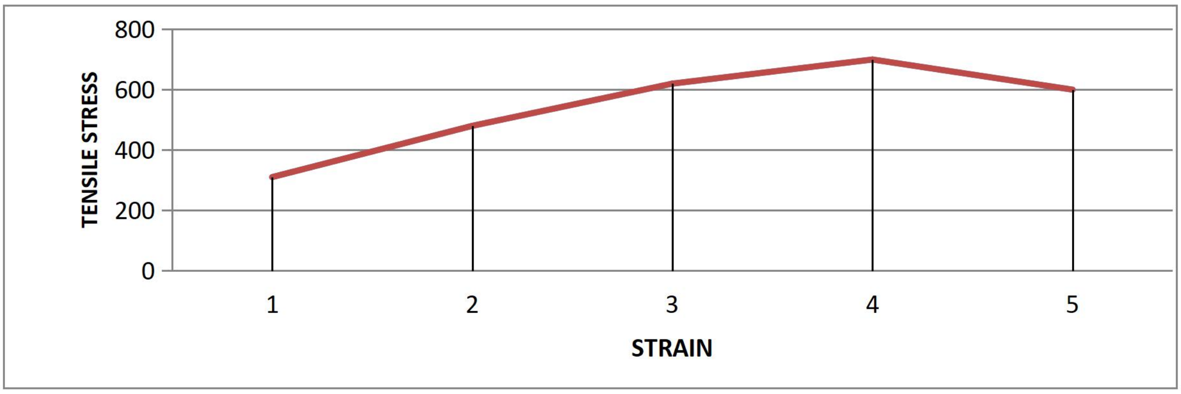


Figure 8: Tensile test (Ti2.0%Cu)

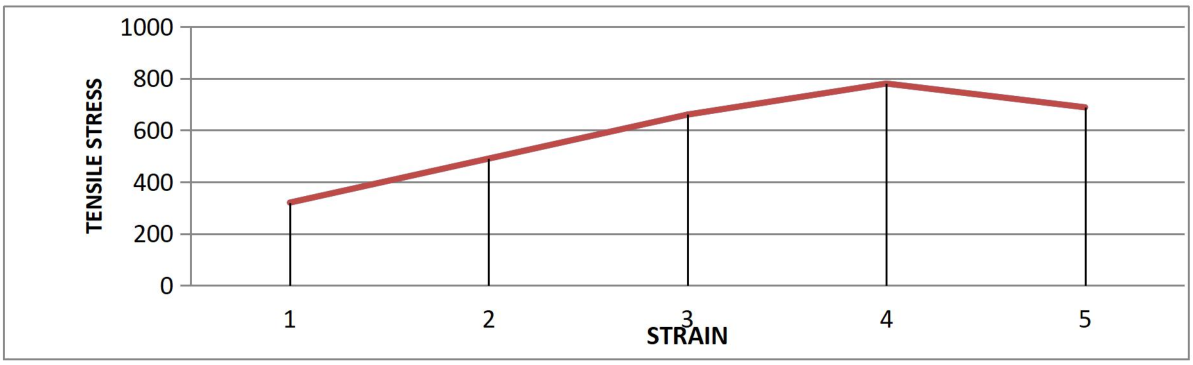


Figure 9: Tensile test (Ti5.0%Cu)

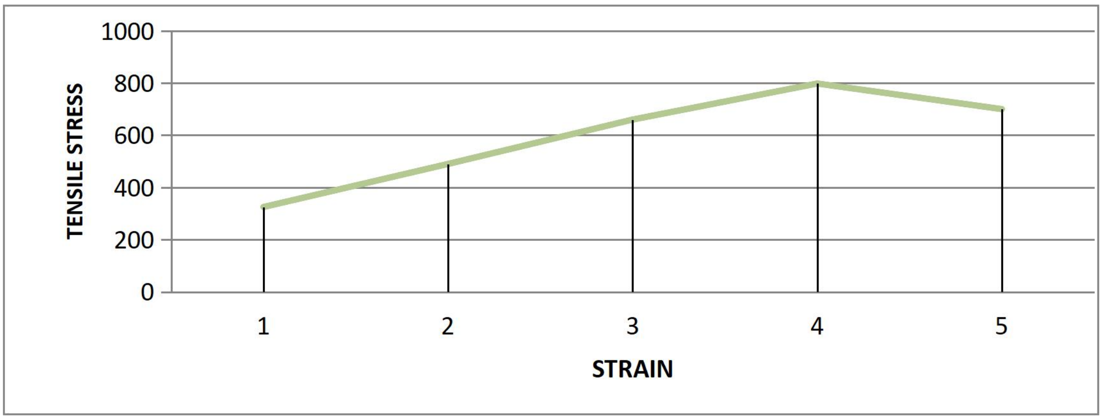


Figure 10: Tensile test (Ti10.0%Cu)

Figure 11: Tensile test (Ti 15.0%Cu)

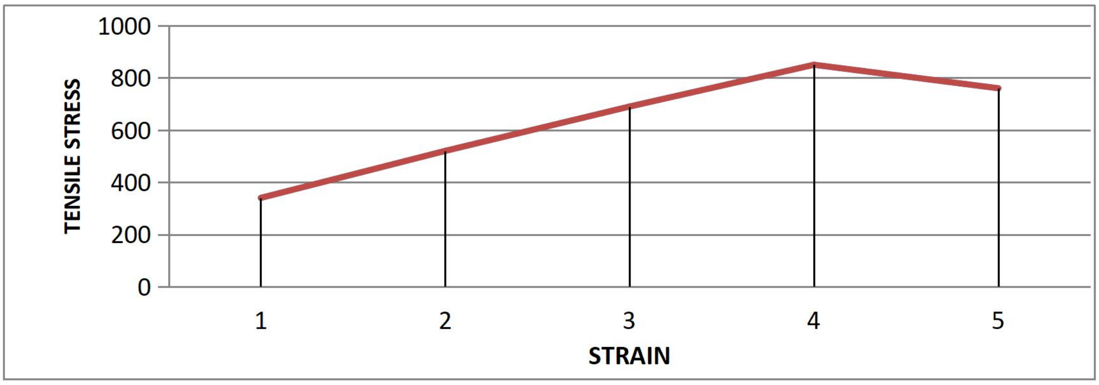


Figure 12: Tensile test (Ti17.0%Cu)

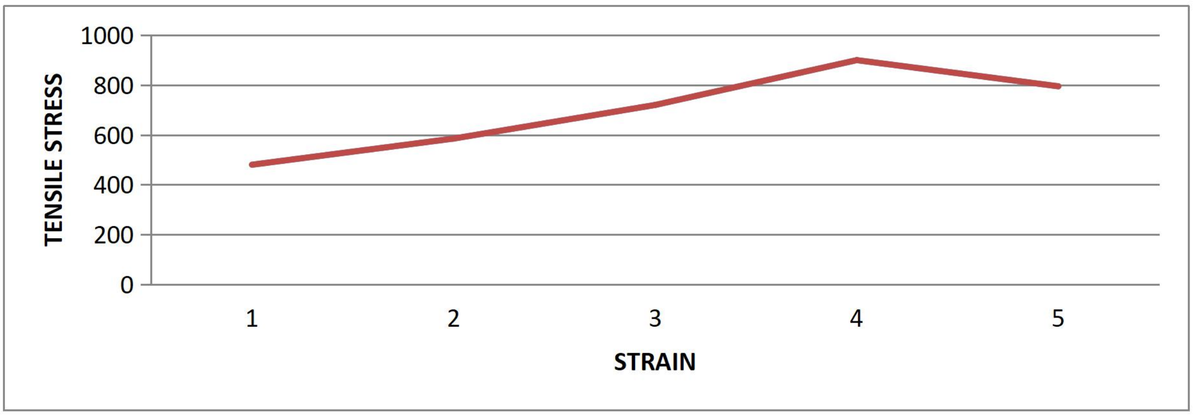


Figure 13: Tensile test(100%Cu)

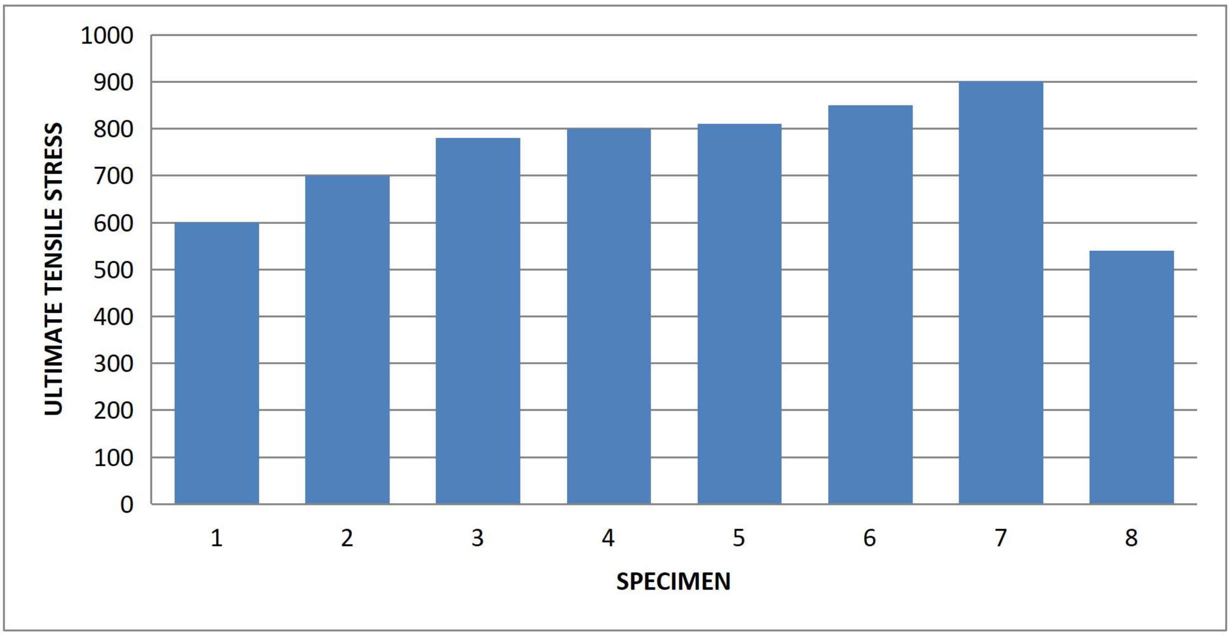


Figure 14: Ultimate tensile Strength

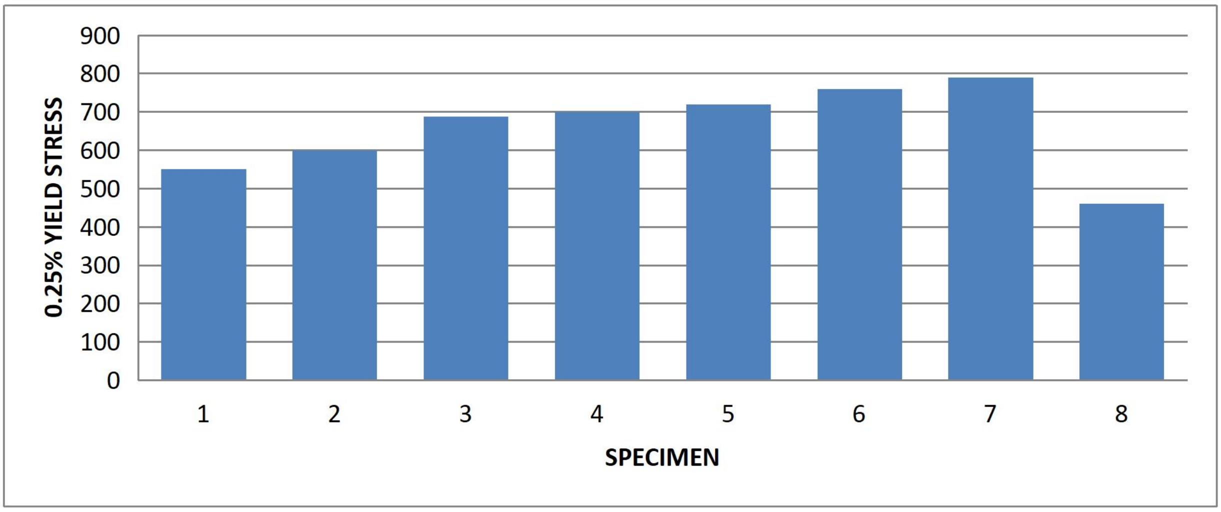


Figure 15: Percentage Elongation

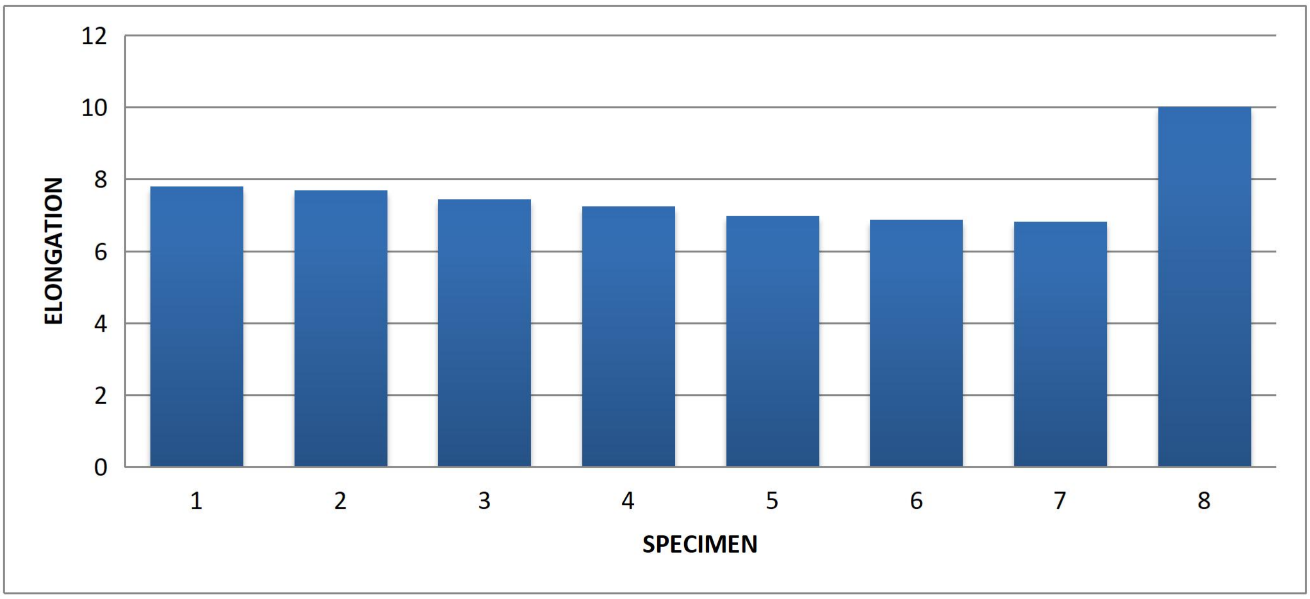


Figure 16: Yield Stress at 0.25%

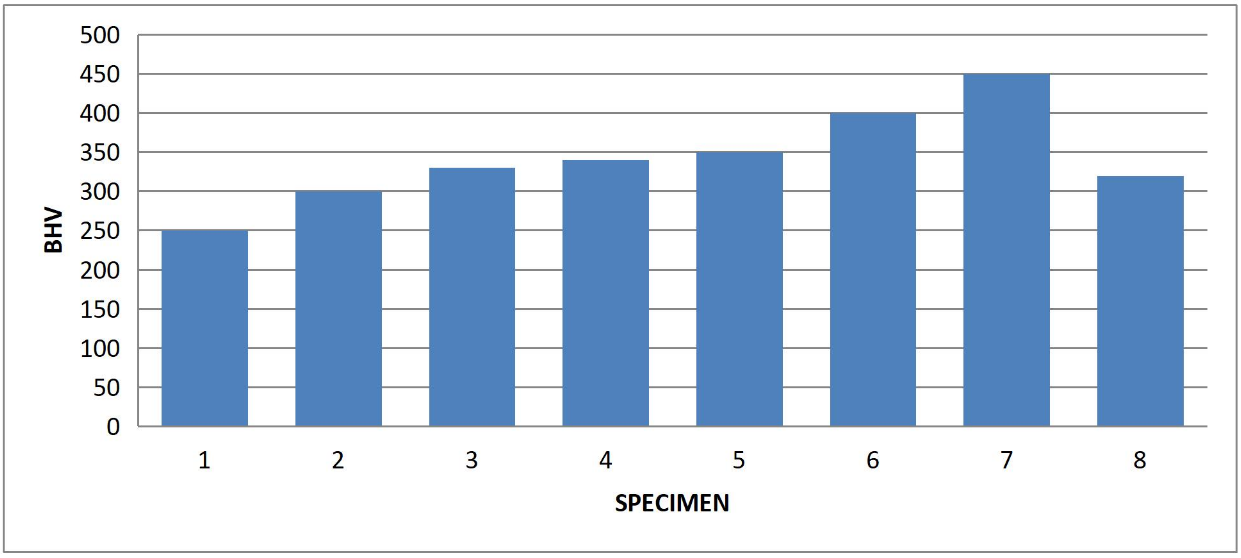


Figure 17: Brinnel Hardness Value (BHV)

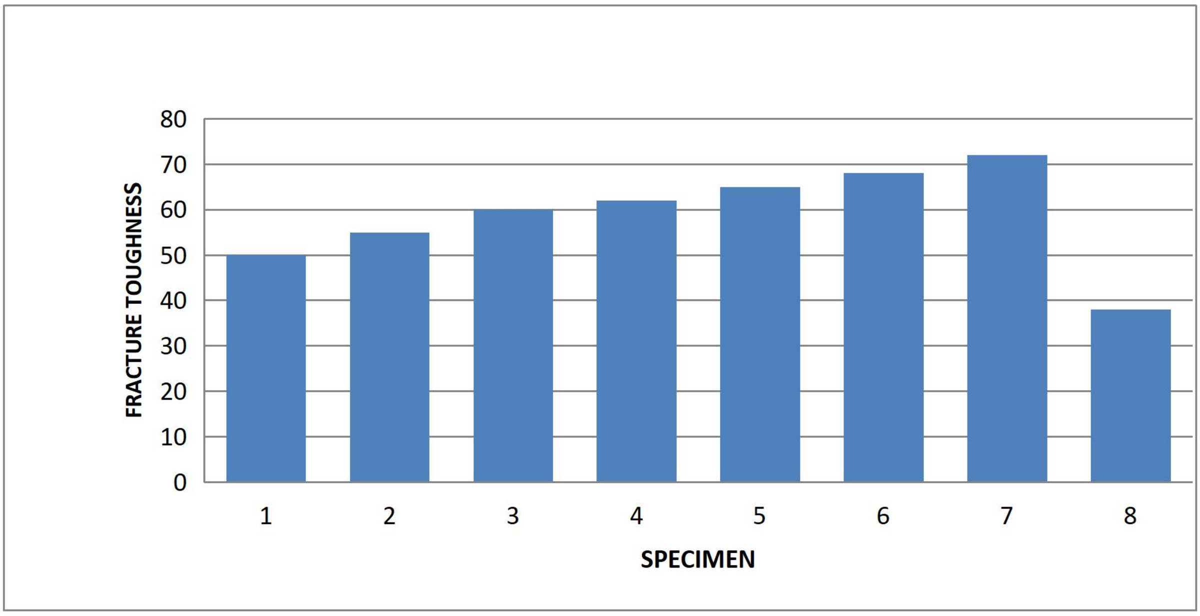


Figure 18: Fracture Toughness

4.3 Discussion

The XRD and SEM microstructure analyses depicted in Figure 4 indicated the formation of Ti_2Cu intermetallic beta phase, with Bcc structure for all the beta phase Titanium copper alloy specimens (Qizh and George,2015). This microstructure stability affected positively the mechanical strength, the microstructure of the Titanium Copper alloy, as shown in Figure 5, indicates that the Copper powder is uniformly distributed within the Titanium matrix, and is an index for good mechanical strength(Congelo and Heiser,1974). Scanning Electron Microscopy (SEM) showed the presence of intermetallic Titanium copper,

Ti_2Cu, which provided the interface for mechanical strength and good biocompatibility properties(Sykaras, 2000; Ezeh 2021). The Elastic Modulus is a measure that determines the resistance of a material to being deformed elastically. This property is essential in biomaterial design, considering the operational load and strains, before surgeries and under service conditions. Strain shows the deformation of a biomaterial under different load conditions, and fatigue investigates the weakening of a material under periodical loads. In biomaterials load deformation during use, there is always an investigation of a possible fracture and subsequent failure in the short or long-term service. Biomaterials must possess a high cycle loading and strength. These very challenging conditions are associated with the hostile in-vivo body environment (Milhov *et al.,* 2009). The specimen with acceptable modulus of elasticity is Ti 17.0% Cu as shown in Table 2 below.

Table 2: Modulus of Elasticity (E) of Specimens

Specimen	Value of E (GPa)
Ti 0.5% Cu	36
Ti 1.0% Cu	37
Ti 2.0% Cu	41
Ti 5.0% Cu	42
Ti 10.0% Cu	44
Ti 15.0% Cu	46
Ti 17.0% Cu	48
Copper (Cu)	30

Where, E= YIELD STRESS/STRAINXFACTOR OF SAFETY (n); n=1.10 factor of safety for biomaterial.

4.4 Regression Model Equations and Statistical Deterministic Validation.

(1) Response Surface Regression: Ultimate Stress versus Ti, Cu

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
61.7267	85.93%	75.38%	*

Regression Equation;

$$\mathbf{ULT = 1051 + 13.2\,Ti - 5.1\,Cu - 0.1689Ti^2} \quad (1)$$

Response Surface Regression: Yield Stress versus Ti, Cu

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
21.0212	**94.87%**	**91.03%**	*

Regression Equation;

$$\mathbf{YIELD} = -1559 + 31.9\,Ti + 21.6\,Cu - 0.1126\,Ti^2 \quad (3)$$

Response Surface Regression: Fracture Toughness versus Ti, Cu

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
3.22114	**95.02%**	**91.29%**	*

Regression Equation;

$$\mathbf{FRA.} = 24 + 1.73\,Ti + 0.14\,Cu - 0.01433\,Ti^2 \quad (4)$$

Response Surface Regression: Brinnel Hardness Value (BHV) versus Ti, Cu

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
29.1618	**86.89%**	**77.06%**	*

Regression Equation;

$$\mathbf{BHV} = 1976 - 6.5\,Ti - 16.6\,Cu - 0.1049\,Ti^2 \quad (5)$$

4.5 Validation of Results

The Minitab software analyses confirmed the statistical Deterministic correlation of the variables (R2) for the parametric experimental analyses as 0.85 < R2 < 0.95. This regressional relationship further confirms the high acceptability of the research results in conformity with the research works of Jin et al (2015) and Erlin et al (2013) on the characterization of Titanium-based binary alloys.

5.0 Conclusion

Many researchers have worked on mono elements, copper(cu), and silver(Ag) materials to function as contraceptive devices, but there is no record of research on the application of binary alloy, specifically beta(β) –phase(Bcc) Titanium copper (TiCu) alloy for gynaecological contraceptive application. Summarily, alloying of the elements(Ti and Cu) in an inert condition, within the temperature of $928^{O}C$ -$1005^{0}C$ achieved a eutectic (BCC) beta phase. Titanium and its alloys in the beta phase domain exhibit microstructure effects of Osseointegration, osteoconduction, and osteoinduction properties of biomaterials. The Osteoinduction is an attribute of Titanium that guarantees the bone healing process with the formation of preosteoblasts, and the reduction of cracks and fractures initiated by the corrosion process, thereby enhancing strength, offered by the acceptable modulus of elasticity index of (Ti17%Cu)**.** This experimental research has provided a knowledge interface in the development of a beta (β) phase biomaterial alloy (Ti17%Cu) with Bcc microstructure and acceptable modulus of elasticity of 48GPa, that are anchored on software designed parametric model equations, thus proferring an improved alternative solution to intractable intrauterine contraceptive challenges of the existing mono element, copper IUDT380 biomaterial.

Declarations

Ethics approval and consent to participate

Not applicable

Consent for publication

Not applicable

Availability of data and material

Not applicable

Competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper

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