Novelty Journals

ISSN 2394-7349

International Journal of Novel Research in Engineering and Science
Vol. 11, Issue 2, pp: (44-58), Month: September 2024 - February 2025, Available at: www.noveltyjournals.com

# EVALUATION OF ANTIBACTERIAL - MICROBIAL PERFORMANCE OF BETA PHASE TITANIUM COPPER ALLOY FOR INTRAUTERINE GYNAECOLOGICAL APPLICATION

[1]Nwogbu Celestine Chidi, [2]Udeh Ubasinachi Osmond, [3]Nwogbu Paul Iloabuchi

Material and metallurgy Department Enugu State University of Science and Technology, Enugu

*DEPARTMENT OF MECHANICAL /PROD. ENGINEERING, CARITAS UNIVERSITY AMORJI-NIKE-NIGERIA

Chemical Engineering Department Enugu State University of Science and Technology, Enugu

**DOI: https://doi.org/10.5281/zenodo.14785074**

**Published Date: 01-February-2025**

*Abstract:* **The research study on the anti-bacterial property of beta phase TiCu alloy was handled by antibacterial test on Eschericha Coli(E,C) and Staphylococcus Aureus(S.A) colonies on the specimens . Research studies indicated that the Ti 17%Cu alloy specimen exhibited excellent antibacterial property, providing a great potential in clinical application for gynaecological implants.The research of antimicrobial/antibiofilm activities of the beta phase contraceptive Titanium Copper alloy (TiCu) against these bacterial species showed an excellent antibacterial effect in vitro, thus inhibiting both Escherichia coli and Staphylococcus Aureus. This research innovated the design and production of Beta((β) phase TiCu alloy specimens in the categories of ; 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 element as the experimental control reference biomaterial. The TiCu alloy specimens were produced by powder metallurgy technique in an inert environment. Experimental investigations and Minitab Software Design analyses on the these specimens for combined antibacterial response in direct improvement of mechanical compatibility (tensile strength, hardness value, fracture toughness,), biocompatibility–cell viability (cytocompatibility), corrosion resistance by Electrochemical Impedance Spectroscopy (EIS), contraceptive response integrity(CRI) and the effect of surface treatment were all carried out in order to establish the suitability of Ti17%Cu alloy as a possible replacement to the existing prototype biomaterial( Cu-T380(IUD).**

*Keywords:* **biomaterials, contraceptive, titanium copper alloy, gynaecological, beta phase, specimens, endometrium, Cu T380IUD, intra uterine.**

## 1. INTRODUCTION

Most metals (Cu ,Ag ,Zn etc) have been reported as being antibacterial agents in metallic matrix.Therefore, elements alloyed with these trace elements are used to develop antibacterial biomaterial metals for orthopaedic, gynaecological and dental implants. Copper element is a prominent material alloyed with other elements inorder to establish antibacterial property. Recently, most metals having eutectic alloy composition are used as biomaterial with antibacterial response (Erlin Zhang, 2013)

Titanium alloy exhibited excellent antibacterial property and corrosion resistance, providing a great potential in clinical application for gynaecological implants.Also 420-CuSS presented excellent antibacterial performance against colonies of bacteria, including,Eschericha Coli (E.coli) and Staphyloccocu Aureus (S. Aureus) , with approximately 99.4% of antibacterial rate. Further studies suggested that 316L SS coated with a TiCu layer (316L/TiCu) showed an effective reduction of 99.9% of Eschericha coli within 12 h 127min (Chen, 2004). In addition, developed alloy containing antibacterial Titanium alloy (Ti5Cu), possess strong antibacterial activity against both Eschericha Coli (E.C) and Staphylococcus Aureus(S.A). The antimicrobial/antibiofilm activities of TiCu alloy(alpha) against the orally specific bacterial species Streptococcus Mutans and Porphyromonas Gingivalis showed an excellent antibacterial effect both in vitro and in vivio, thus inhibiting Escherichia coli. Ti5%Cu have Strong antibacterial activity against both Escherichia coli and Staphylococcus Aureus (Hong, 2005) .

Biomaterial research reports established that human tissue is mainly organised 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 immotility 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. The application of metals and alloys for biomedical contraceptive within the endometrium host environment is a research innovation, not yet explored fully . Williams(2013), a biomedical expert, reported that heavy metal ions possess spermicidal properties, and anchored this research finding on the effects of $Cu^{2+}$, $Zn^{2+}$, $Fe^{2+}$,$Pb^{2+}$, $Cd^{2+}$, $Mn^{2+}$,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 body centered cubic system(bcc) in beta(β) phase, because it possesses high passivity and regenerative properties i.e ability to repair itself and form protective covering, with dense oxide film(Coating,2003). It is usually considered for biomaterial, and the low young modulus is very close to that of the bone (williams, 2013), which disallows the stress shielding effect associated with biomaterials of high modulus of elasticity, common to alpha (α) and alpha+beta (α +β) phases. Titanium alloys are differentiated into three metallurgical groups, which are; alpha (α), Beta (β )and alpha+beta. Research has shown that copper phase stabilizes Titanium alloys, and these are qualitatively used in gynaecological biomaterial application, with very low modulus of Elasticity (which is below the α and α + β phase) , and very close to human femoral bone modulus of elasticity of between 38 – 40GPA(Sammons,2003).Titanium and its alloys in beta phase domain exhibit microstructure effects of Osseointegration, osteoconduction and osteoinduction properties of biomaterials .The Osteoinduction is an attribute of Titanium which guarantees bone healing process with formation of prosteoblasts, and the reduction of cracks and fractures initiated by corrosion process(Sutter and Bonni,2005).

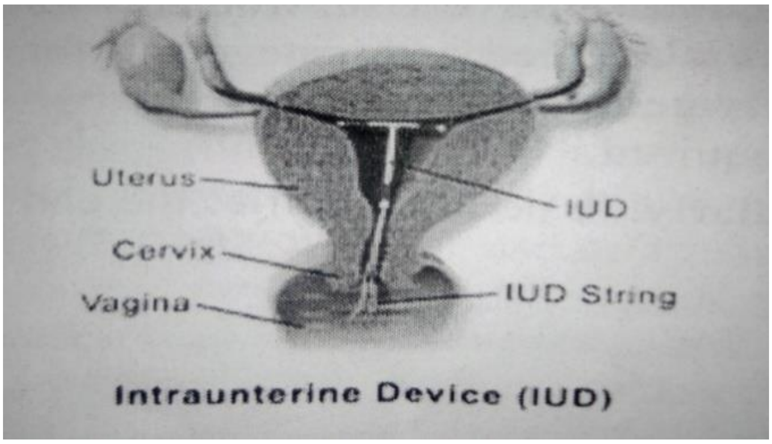



**Figure 1: Insertion of IUD T380 in the Endometrium Ref; Kalpana Gupta (2009)**

## EXISTING PROBLEMS IN INTRAUTERINE CONTRACEPTIVE BIOMATERIAL

The problems of existing Intrauterine implants(CuT380) are basically 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 are due to corrosion and wear thereby influencing the mechanical properties of the biomaterial, and usually lead to mechanical failure(Gilbert,1993).

Novelty Journals

ISSN 2394-7349

International Journal of Novel Research in Engineering and Science
Vol. 11, Issue 2, pp: (44-58), Month: September 2024 - February 2025, Available at: www.noveltyjournals.com

**MATERIAL DESIGN OF CYLINDRICAL SPECIMENS BY PHASE DIAGRAM**

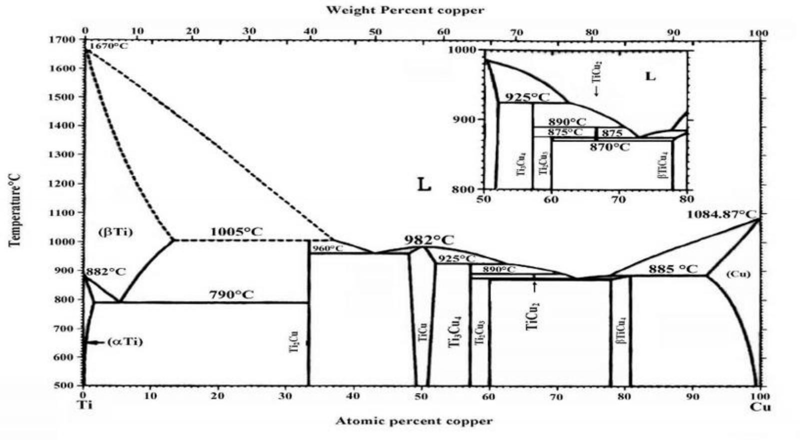



**Figure 2: Phase Diagram of Ti-Cu Alloy (courtesy, Good fellow Inc, USA)**

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 the inert environment at the temperature of $1005^0$C (Udeh,2021) .

**PREPARATION OF SPECIMENS BY POWDER METALLURGY**

The specimens were prepared by Powder metallurgy approach by;.

i. Copper: commercial pure copper powder named(cp-Cu) were used for the alloying, and also as specimen for the control reference experimental biomaterial.

ii. Titanium-copper alloy(TiCu) specimen preparations , with prepared Titanium powder and Copper powder (99.5% pure) of different percentage weight compositions were 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).

The Titanium powder and copper powder were each weighed out differently, and ball milled differently for 4-7 hrs, and then were pressure compacted upto 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 mins, and allowed to cool in furnace to room temperature of $30^{O}$C . The specimens of diameter 30mm and a thickness of 2.5cm were sliced-off from the TiCu specimens for the various tests (Udeh,2021).

**Fig 3: Developed beta phase Titanium copper specimens and copper specimen**

International Journal of Novel Research in Engineering and Science
Vol. 11, Issue 2, pp: (44-58), Month: September 2024 - February 2025, Available at: www.noveltyjournals.com

**EXPERIMENTAL TESTS (DESIGN APPROACH)**

The experimental tests conducted, within this biomaterial research work are as follows;

(1) Electrochemical Corrosion resistance test (EIS)

(2) Biocompatibility (cell viability / cell proliferation) test

(3)Mechanical compatibility test (Tensile, Hardness, Fracture Toughness tests)

(4) Antiseptic /Microbial test

(5) Invitro Contraceptive response test

(6) Microstructure and XRD Examination

**Electrochemical Impedance Spectroscopy Test for Corrosion (EIS)**

The electrochemical impedance spectroscopy experiments were conducted with the use of two fluids (Simulated Body Fluids) which are simulated to be identical to human fluids, controlled at 7.1 PH value, using Hanks solution and Ringers solution, This test is carried out at approximtely $37^0$c ,,with beakers containing the specimens. The open circuit potential (ocp) was measured for 1 hour(3600secs) of immersion of the specimens. The electrochemical impedance spectroscopy test for the specimens were conducted at a room temperature condition of $37^0$c, with scan from - 190mv $v_s$ocp(open circuit potential) to + 1250 mv $v_s$ocp at a scan rate of 0 .5mv/s . Impedance measurements were performed at +10mv amplitude upto 1hr, recording the electrochemical impedance spectroscopy spectra at approximately every 60secs for the measurement tests .

The corrosion rate is,

$$V = \frac{MI}{nF} \qquad (3.4)$$

Where,

M = molar mass of Titanium copper or Copper element

I = Average corrosion rate

F= Faradays Corrosion density

n = Valency of element

**Table 1: Hanks Solution**

| Substance | Composition (g $L^{-1}$) |
|---|---|
| NaCl | 8.0 |
| KCl | 0.4 |
| $NaHCO_3$ | 0.35 |
| $NaH_2PO_4.H_2O$ | 0.25 |
| $Na_2HPO_4.2H_2O$ | 0.06 |
| $CaCl_2.2H_2O$ | 0.19 |
| $MgCl_2$ | 0.19 |
| $MgSO_4.7H_2O$ | 0.06 |
| Glucose | 1.0 |
| PH | 6.9 |

**Ref; (Aragon P.J,2009)**

**Table 2: Ringers solution**

| | |
|---|---|
| NaCl | 8.69 |
| KCl | 0.30 |
| $CaCl_2$ | 0.48 |
| PH | 6.4 |

**(Ref; Aragon P.J, 2009)**

**The biocompatibility tests of the specimens** were investigated by the process of Cellular attachment (adhesion) and Mitochondrial activity (MTT) test of human osteoblast-derived cells compared to the bare substrate. .The samples placed in 8 cultured plates and cells each, were plated and left to adhere for 3.5hrs, and after this duration, 500 microlitres of culture meduim were added.

## 2. MECHANICAL COMPATIBILITY TEST

**Microhardness test:**

Microhardness test was handled by first polishing the specimens, and point of identation mark occurrence on their surfaces recorded by the identation 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.

**The Tensile test**

The tensile and percentage elongation tests on the specimens were handled with the universal testing machine.The machine has a loading frame, and a controller plotter, with progressive strain rates of 0.01/sec, 0.015/sec, 0.020/sec, 0.025, 0.030/sec .The loads versus strains on each specimen were recorded.

**Fracture Toughness Test:**

Fracture toughness test on the specimens progresses with surface polishing to remove any identation, 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.

**Antiseptic Microbial Test**

The antiseptic microbial tests on these samples,using colonies of staphylococcus Aureus and Eschericha Coli in plated and incubated media at 6-8 hours of incubation with the antibacterial rate, R, is calculated from the equation;

$$R = (N_{conrol} - N_{sample})/N_{conrol} \% \quad (3.5)$$

Where,

$N_{control}$ = average number of bacteria on the control dish

$N_{sample}$ = average number of bacteria on the sample dish

R>99% denotes that sample has strong antibacterial property

R >90% denotes that sample has simple antibacterial property

**The invitro contraceptive (spermicidal) test** is conducted by using the estimation of motility of the spermatozoa collected and their metabolism.

## 3. PREPARATION OF SPERMATOZOA

The Semen were collected from the laboratory. The semen is maintained at $37^0$c for 15 to 30minutes .The spermatozoa were mixed by adopting gentle agitation. The spermatozoa is prepared by washing the spermatozoa free of seminal plasma and diluted with 5 volumes of free calcium Kreber Ringer phosphate (KRP) buffer (0.12m NaCl. 0.005m KCl, 0.0124m Mg $So_4$,.0.016 m Soduim Phosphate, PH 7.4 at $37^0$c, and then centrifuged at 740 xgl ($r_{av}$=17cm) for period of ten(10) minutes at $20^0$c.The aliquots are then removed and the spermatozoal suspension collected for use,with each specimen placed into each container.

**Estimation Test of Motility**

The use of flasks containing 1.01 + 0.1 x $10^8$ spermatozoa, using the 300 Sq mm of the specimens to be tested and 3Mm glucose in KRP buffer were incubated for 3hrs with constant shaking at $37^O$C in a water bath , with Spermatozoa added at intervals of 30,60,120 and 180 minutes.

**Evaluation Test of Metabolism**

A special container having small flasks , and in which manometer was immersed in a thermostatically controlled bath assisted in measuring the oxygen consumption of the spermatozoa.Approximately, 1.01 + 0.1 x $10^8$ spermatozoa were added to each flask,which contained 3Mm glucose(0.5x$10^{-6}$gm of glucose), and the 300Sqmm of the specimens under test.

**OBSERVATIONS;**

Direct electronic microscopic observations were used for the evaluations.

Novelty Journals
ISSN 2394-7349
International Journal of Novel Research in Engineering and Science
Vol. 11, Issue 2, pp: (44-58), Month: September 2024 - February 2025, Available at: www.noveltyjournals.com

## 4. RESULTS AND DATA ANALYSIS

In the development of Ti x%Cu (x= 0.5,1.0,2.0,.5.0,10.0,15.0 and 17.0 %) alloy specimens and copper element as reference control experiment, there is a research trend employing engineering Minitab design methodology for investigation, of designed experimental test on parameters such as biocompatibility status ,mechanical strength, corrosion resistance rate and antibacterial properties in order to establish the suitability of these specimens to function effectively as contraceptive biomaterial within the endometrium (Ullman and Hammerstein,1993).

**MECHANICAL TESTS**

**TENSILE STRESS TEST AND PERCENTAGE ELONGATION RESULTS**

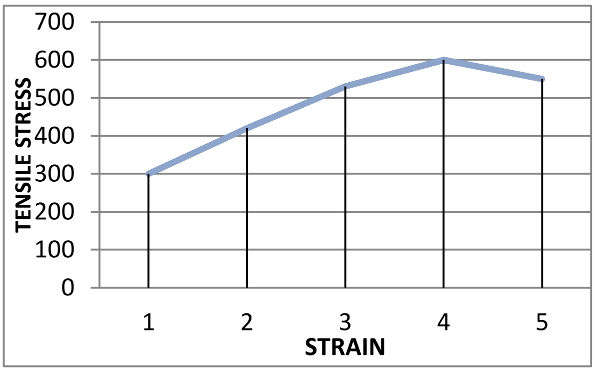
The Tables below show the results

**PLOTS OF TENSILE STRESS**

Plots of Tensile stress versus strain for the various specimens are shown below;

**(1) Ti 0.5%Cu, (2)Ti 1.0%Cu, (3)Ti 2.0% Cu, (4)Ti 5.0%Cu,**

**(5) Ti 10.0%Cu, (6)Ti 15.0%Cu, (7)Ti 17%Cu and (8) 100%Cu**



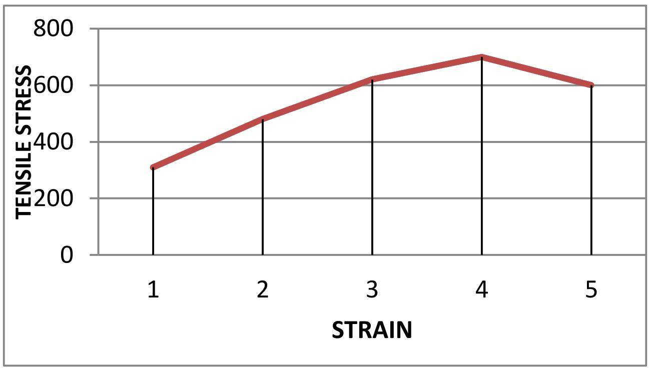
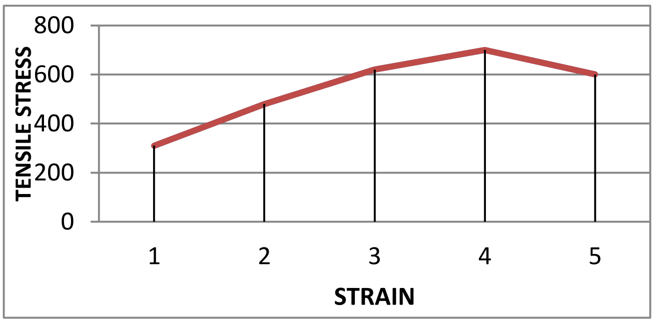




**Fig 4: Tensile test(Ti0.5%Cu)** **Fig 5: Tensile test(1.0%Cu)**



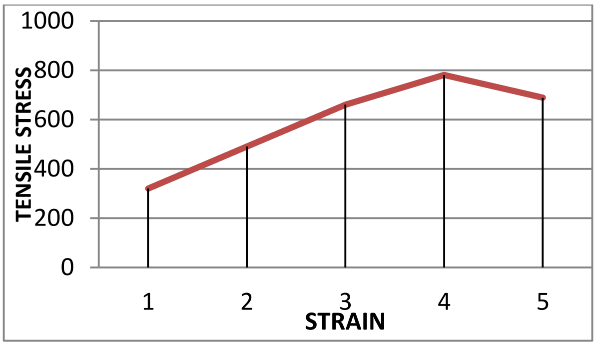




**Fig 6: Tensile test (Ti2.0%Cu)**

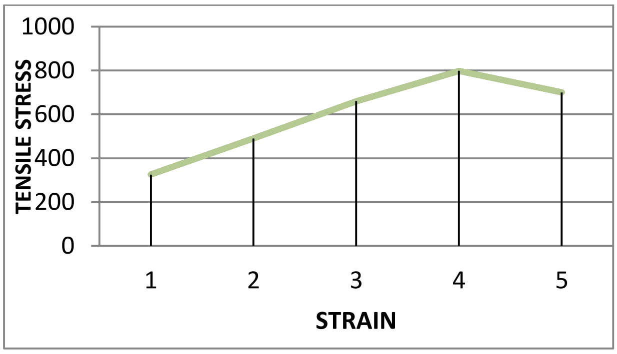




**Fig 7: Tensile test (Ti5.0%Cu)** **Fig 8: Tensile test (Ti10.0%Cu)**

International Journal of Novel Research in Engineering and Science
Vol. 11, Issue 2, pp: (44-58), Month: September 2024 - February 2025, Available at: www.noveltyjournals.com

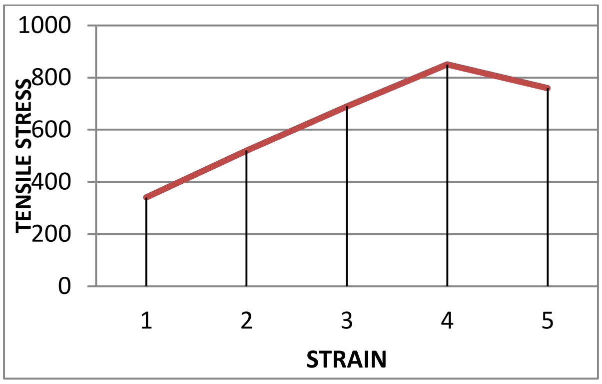



**Fig 9: Tensile test(Ti 15.0%Cu)**

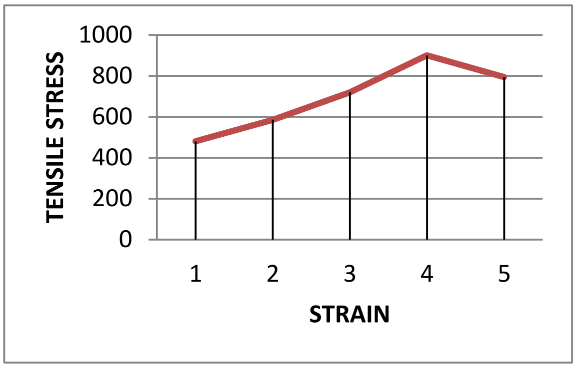



**Fig 10: Tensile test(Ti17.0%Cu)**

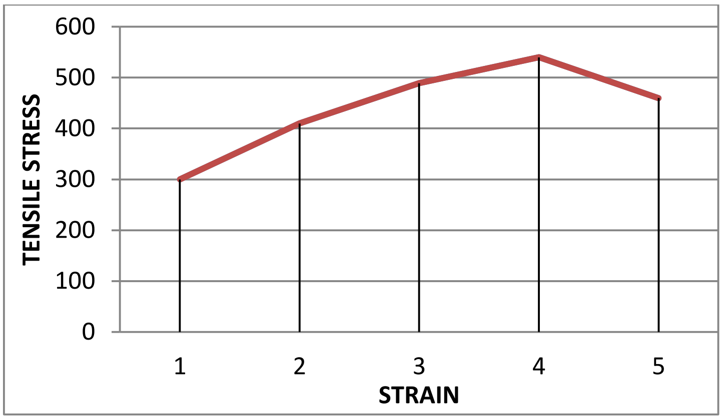



**Fig 11: Tensile test(100%Cu)**

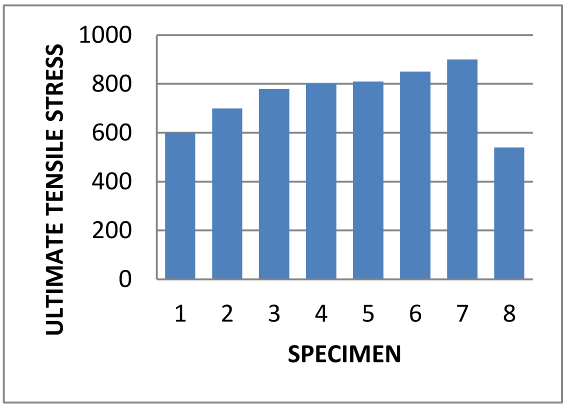



**Fig 12: Ultimate tensile Strength**

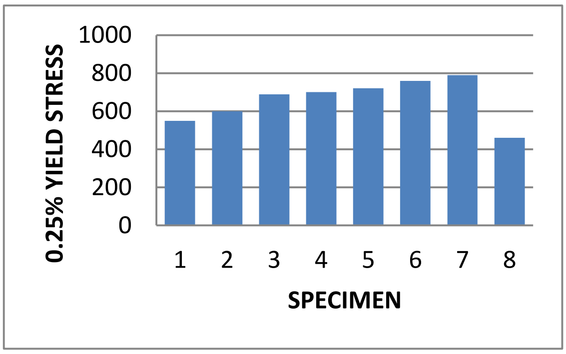



**Fig 13: Percentage Elongation**

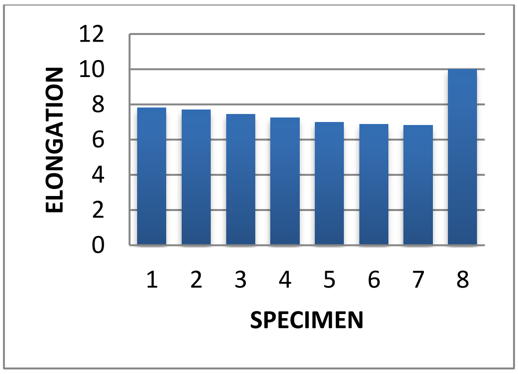



**Fig 14: 0.25% Yield Stress**

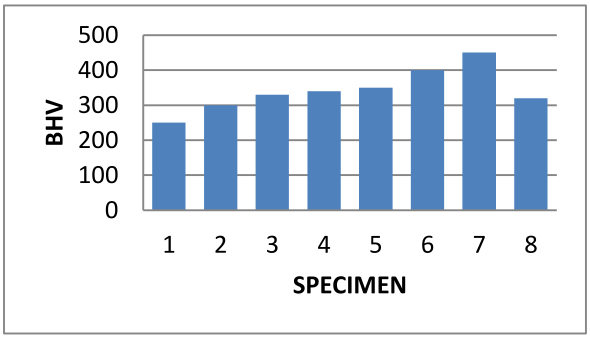



**Fig 15: Brinnel Hardness Value (BHV)**

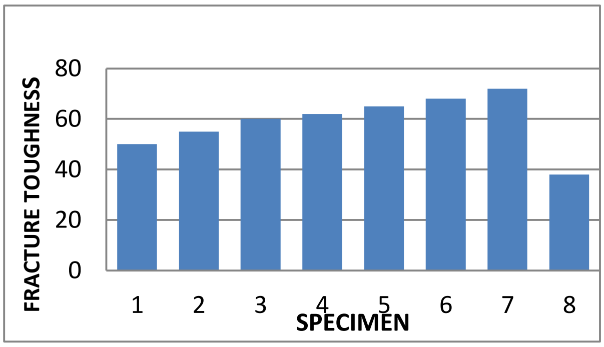


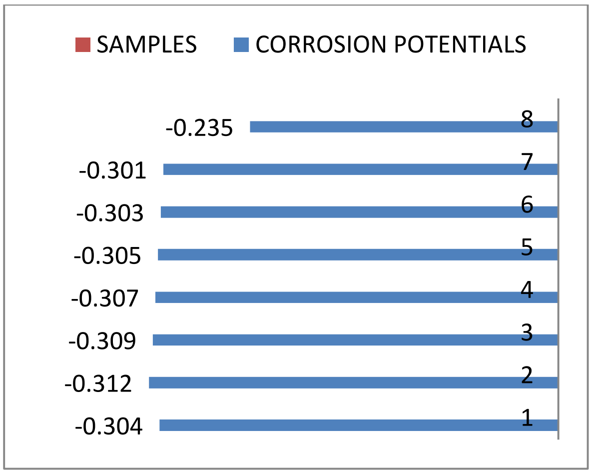

**Fig 16: Fracture Toughnes**

International Journal of Novel Research in Engineering and Science
Vol. 11, Issue 2, pp: (44-58), Month: September 2024 - February 2025, Available at: www.noveltyjournals.com

**4.3 CORROSION TEST RESULTS.**

The corrosion potential of the specimens showed that Ti 17%Cu have value of -0.304 While copper indicated a corrosion potential of -0.243. .

***HANKS SOLUTION SIMULATED BODY FLUID (SBF)*** **CORROSION RATE (HANKS)**




**+Fig 17: Corrosion Potential**

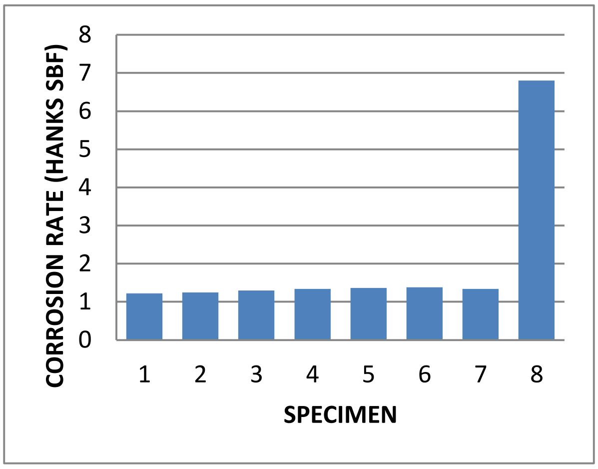



**Fig 18: Corrosion Rate (Hanks)**

**PLOT OF CORROSION RATE (RINGERS)**

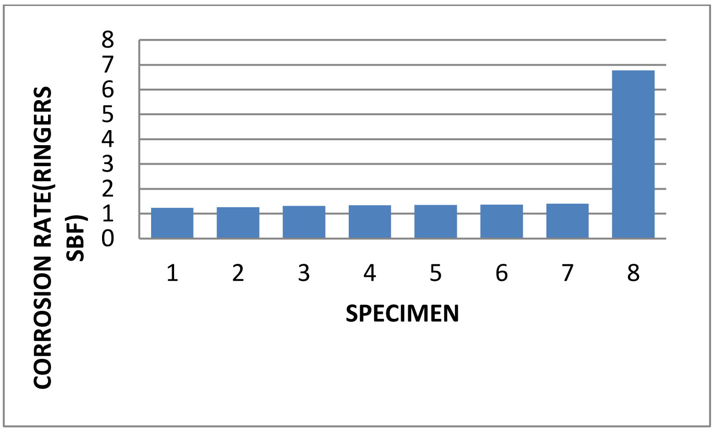



**Fig 19: Corrosion Rate (Ringers)**

**BIOCOMPATIBILITY TEST RESULT**

The biocompatibility – cell viasbility test showed that Ti 17%Cu has 155% viability while copper has 54% cell viability status. The Copper ion release is 0.029mg/Litre for Ti 17%Cu , while copper has ion release of 0.038mg/litre

**CELL CYTOTOXICITY. COPPER ION RELEASE TEST**

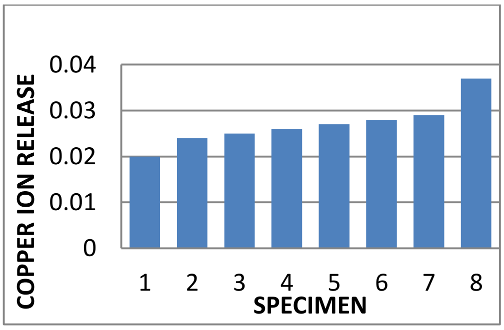



**Fig 20: Copper ion Release (Cytotoxicity)**

**International Journal of Novel Research in Engineering and Science**
Vol. 11, Issue 2, pp: (44-58), Month: September 2024 - February 2025, Available at: www.noveltyjournals.com

**ANTIMICROBIAL - ANTISPETIC TEST RESULTS**

The antiseptic microbial test on the specimens, in the cultured environment of each of Staphylococcus Aureus (S.A) and Escherisha Coli(E.C) colonies, indicated progressive antibacterial action. with increase in the percentage composition of copper. .

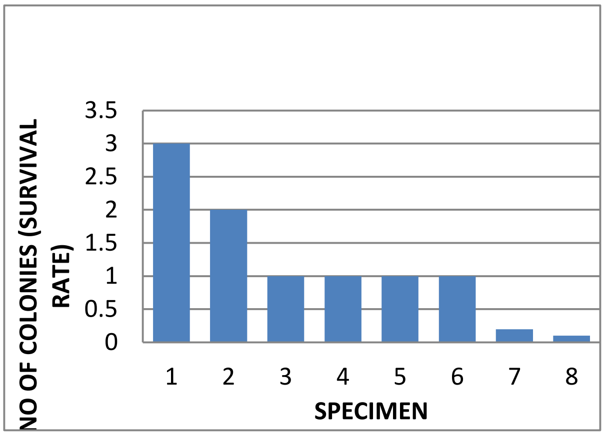



**Fig 21: Staphylococcus Aureus (S.A)**

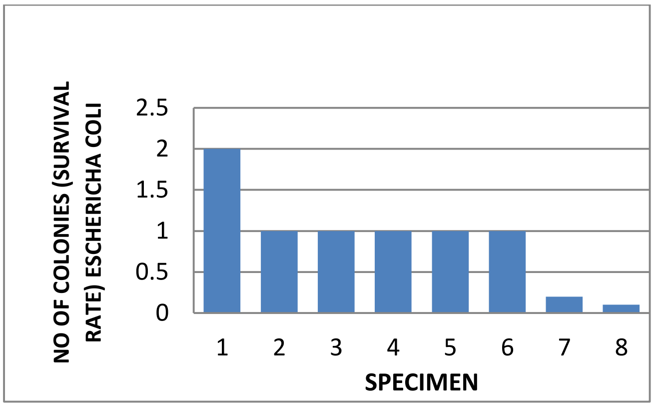



**Fig 22: Escherisha Coli(E.C)**

**INVITRO CONTRACEPTIVE RESPONSE TEST RESULTS**

On the Contraceptive response test, the research initiated invitro simulative approach of percentage Motility rate and Glycolytic Metabolism assessment.

**PERCENTAGE MOTILITY RATE**

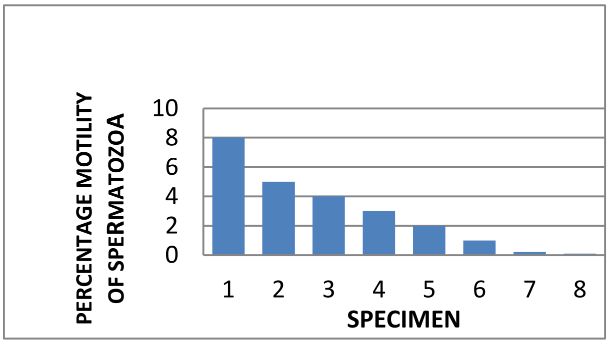



**Fig 23: Invitro Contraceptive (Percentage Motility Rate)**

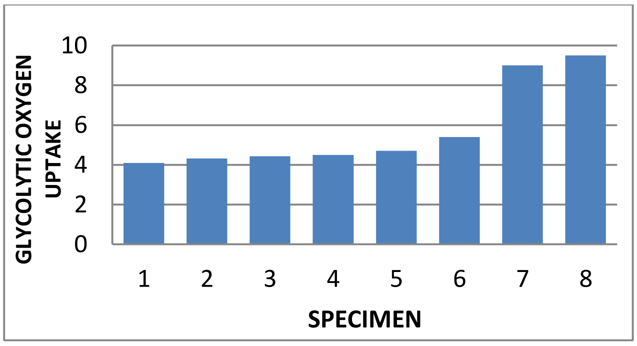



**Fig 24: Glycolytic Oxygen Uptake(invitro Contraceptive)**

**TABLE 3: Experimental Results for Minitab Software Design Analysis.**

**RESULTS FOR MINITAB ANALYSIS**

| SAMPLES | | Ult | YIELD | FRA. | BHV | COIR | Ecorr | VIA | MOT | GLY |
|---|---|---|---|---|---|---|---|---|---|---|
| *TI* | *CU* | *MPA* | *MPA* | *MPA $M^{0.5}$* | *VALUE* | *Mg/cm2yr* | *-* | *%* | *%* | *%* |
| ***99.5*** | ***0.5*** | ***600*** | ***550*** | ***50*** | ***250*** | ***1.22*** | ***-0.317*** | ***166*** | ***8*** | ***4.1*** |
| 99.0 | 1.0 | ***700*** | ***600*** | ***55*** | ***300*** | ***1.25*** | ***-0.315*** | ***165*** | ***5*** | ***4.32*** |
| 98.0 | 2.0 | ***780*** | ***688*** | ***60*** | ***330*** | ***1.30*** | ***-0.313*** | ***164*** | ***4*** | ***4.44*** |
| 95,0 | 5.0 | ***800*** | ***700*** | ***62*** | ***340*** | ***1.34*** | ***-0.310*** | ***160*** | ***3*** | ***4.5*** |
| 90.0 | 10.0 | ***810*** | ***720*** | ***65*** | ***350*** | ***1.36*** | ***-0.308*** | ***158*** | ***2*** | ***4.7*** |
| 85.0 | 15.0 | ***850*** | ***760*** | ***68*** | ***400*** | ***1.38*** | ***-0.305*** | ***156*** | ***1*** | ***5.4*** |
| 83.0 | 17.0 | ***900*** | ***790*** | ***72*** | ***450*** | ***1.40*** | ***-0.304*** | ***155*** | ***0.2*** | ***8.2*** |
| 0.0 | 100,0 | ***540*** | ***460*** | ***38*** | ***320*** | ***6.8*** | ***-0.242*** | ***54*** | ***0.1*** | ***9.5*** |

**8 (EIGHT) SAMPLES( Ti-*TITANIUM %*, *Cu, = COPPER%)***

***Note:* VALUED PARAMETERS**

ULT -ULTIMATE TENSILE STRENGTH, YIELD– 0.25% YIELD STRENGTH, FRA - FRACTURE TOUGHNESS, BHV-BRINNEL HARDNESS VALUE, COIR- COPPER ION RELEASE RATE, Ecorr – CORROSION POTENTIAL,VIA-BIOCOMPATIBILITY VIABILITY PERCENT,MOT- SPERMATOZOA MOTILITY RATE, GLYM- GLYCOLYTIC METABOLISM.

**TABLE 4: Modulus of Elasticity, E , of Specimen**

| 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 (biomaterial)

**Regressional 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;

**ULT = 1051 + 13.2 Ti - 5.1 Cu - 0.1689Ti²** ($ULT = 1051 + 13.2\,Ti - 5.1\,Cu - 0.1689\,Ti^2$)

(2)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 ;

$$YIELD = -1559 + 31.9\,Ti + 21.6\,Cu - 0.1126\,Ti^2$$

(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;

$$FRA. = 24 + 1.73\,Ti + 0.14\,Cu - 0.01433\,Ti^2$$

(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;

**BHV = 1976 - 6.5 Ti - 16.6 Cu - 0.1049 Ti$^2$**

(5)Response Surface Regression: COPPER ION RELEASE RATE versus Ti, Cu

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| **0.0017480** | **92.72%** | **87.27%** | ***** |

Regression Equation ;

**COIR = 0.168 - 0.00114 Ti - 0.00131 Cu - 0.000003 Ti$^2$**

(6)Response Surface Regression: Ecorrosion Potential, versus Ti, Cu

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| **0.0043111** | **97.24%** | **95.16%** | ***** |

Regression Equation ;

**Ecorr = 0.020 - 0.00556 TI - 0.00263 CU + 0.000027 TI*TI**

(7)Response Surface Regression: Viability-Biocompatibility, versus TI, CU

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| **9.95772** | **94.95%** | **91.17%** | ***** |

Regression Equation;

**VIA = -581 + 11.5 Ti + 6.3 Cu - 0.04769 Ti$^2$**

(8)Response Surface Regression: Motility Rate, versus Ti, Cu

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 1.36955 | 85.34% | 74.35% | * |

Regression Equation ;

**MOT = -84 + 0.53 Ti + 0.84 Cu + 0.00369 Ti$^2$**

(9)Response Surface Regression: Glycolytic metabolism Rate; GLYM versus Ti, Cu

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| **1.02616** | **85.60%** | **74.80%** | ***** |

Regression Equation;

**GLY = 54 - 0.36 Ti - 0.45 Cu - 0.001428 Ti$^2$**

International Journal of Novel Research in Engineering and Science
Vol. 11, Issue 2, pp: (44-58), Month: September 2024 - February 2025, Available at: www.noveltyjournals.com

**XRD AND SEM EXAMINATIONS**

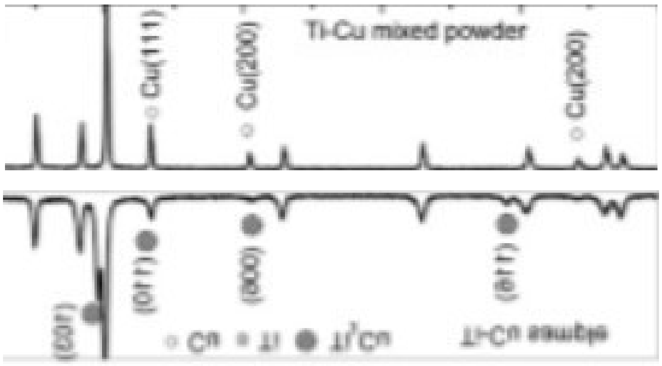



**Fig 25:** ***XRD PATTERN OF Ti 17% Cu AND COPPER FLEMENT***

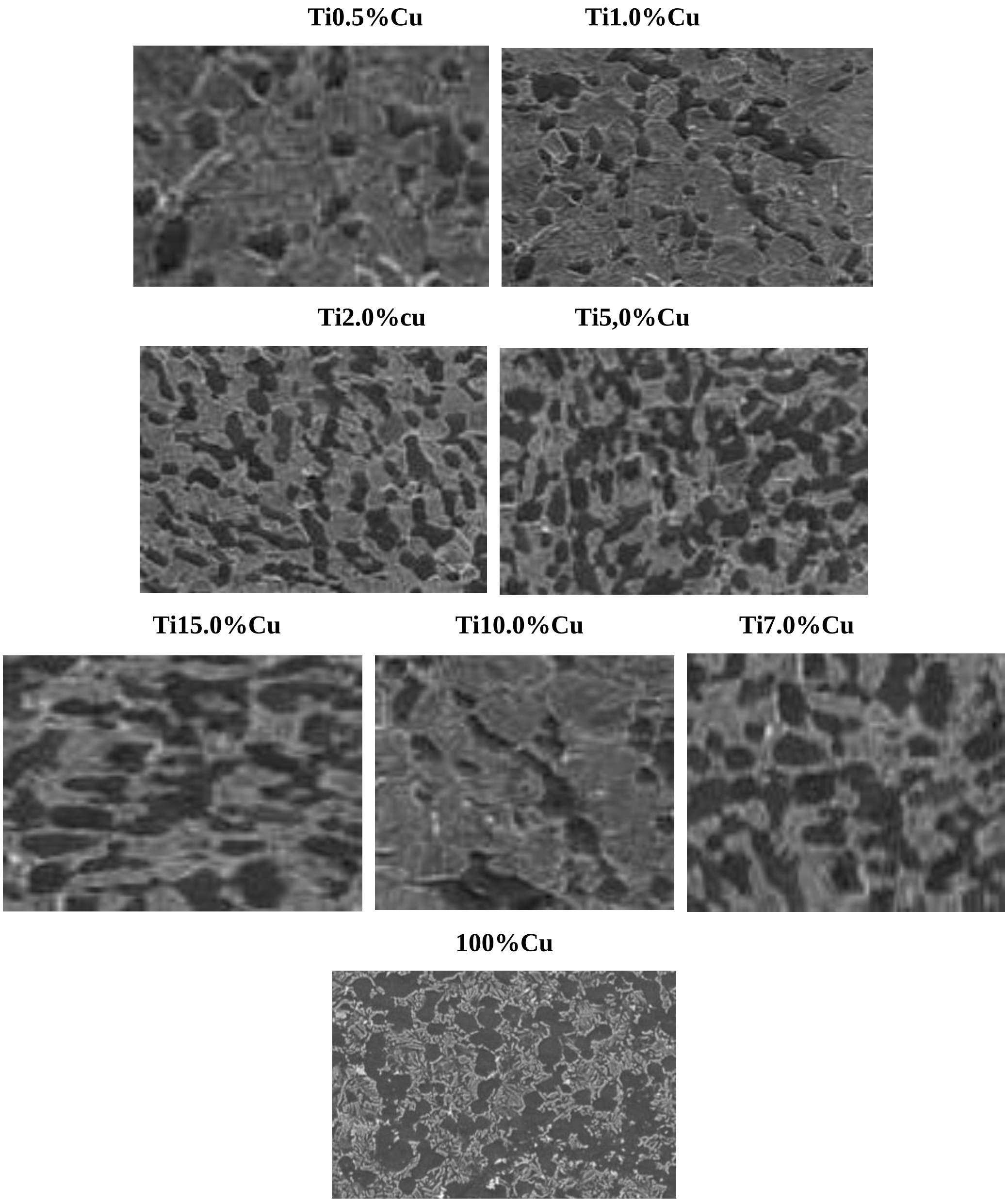



**Fig 26: SEM MICROSTRUCTURE EXAMINATION**

## 5. DISCUSSION

### DEVELOPMENT OF SAMPLES (TiCu) BY POWDER METALLURGY

The development and analysis of the beta phase TiCu alloy (Bcc)specimens is innovated, using the highlighted characterized parameters which influenced the acceptability of the researched alloy, Titanium Copper alloy(Paul and Ronald, 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, copper, at temperature of 928ºC- 1005ºC to form Titanium Copper alloy,there is a metallurgical phase transformation to Beta phase with Body centered cubic structure(Bcc) (Udeh,2021) . .

### MICROSTRUCTURE EXAMINATIONS AND MECHANICAL STRENGTH

The XRD and SEM microstructure analyses 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 microsructural stability affected positively the mechanical strength, .The microstructure of the Titanium Copper alloy, as shown in figure 26, indicates that the Copper powder is uniformly distributed within the Titanium matrix, and is an index for good mechanical strength(Congelo and Heiser,1974).. The 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).

### CORROSION RESISTANCE AND SURFACE FILM REGENERATION

Kobyashi et al (1988) and Fontana (1986) in similar research concluded that Titanium alloys when tested in both simulated body fluids(SBF) of Hanks solution and Ringers solution have very low corrosion rate . Sutter T. and Boni H.(2005) maintained that corrosion resistance of biomaterials, as in Ti17%Cu alloy, is best investigated using Electron Impedance Spectroscopy (EIS), Gonzalez and Mirza(2004) collaborated the research result on these TiCu alloys and confirmed that the low corrosion rate is due to the regenerative oxide film layer which forms protective film and prevents biological attacks by the plasma contents of Oxygen in human system.. .

### BIOCOMPATIBILITY

Chen (2018) research on biomaterial applications confirmed that TiCu alloys ,as in the Ti17%Cu alloy , have very tolerable copper ion release which gives acceptable cell compatibility and viability to these biomaterials. The biocompatibility of the TiCu alloy ,being evaluated by the principles of adhesion viability / proliferation assays , showed that it agreed with the research of Zielske et al 1974 which recorded high cell affinity of beta phase Titanium alloys ,as in Ti17Cu%,, within the interacting biological human tissues. .

### ANTIBACTERIAL AND ANTISEPTIC PROPERTY

Ali (2004) proved that Ti alloys ,as shown in the case of results of Ti17%Cu alloy, exhibited excellent antibacterial microbial quality performance against Eschericha Coli (E.Coli) and Staphyloccocus Aureus (S,A),with an approximate 99,9% antibacterial rate. The normal vaginal system is reported to host urinogenital bacteria and cause subsequent infections .The vaginal flora is disrupted by the presence of antibacterial biomaterial which has microbial and antiseptic properties (Kalpana,2009). The Ti17% Cu response to the most common genital infections in the endometrium of staphylococcus aureus (S.A) and Eschericha coli (E.C) showed inhibition of the colony formation of these microbes, thereby providing good antiseptic qualities. .

### INVITRO CONTRACEPTIVE RESPONSE

Ahti kosonen (1978) results agreed with TI17%Cu alloy and proved that the copper ion release created immotile environment within the endometrium, that inhibited the fertilization of the ovum by the immotile spermatozoa. Zipper (1969) analysis,as in TI17%Cu alloy, showed the effect of copper ion release which inhibited spermatozoan movement in the endometrium that creates infertility and contraceptive action.

### VALIDATION OF RESULTS.

The Minitab software analyses confirmed the statistical Deterministic correlation of the variables ($R^2$**)** for the parametric experimental analyses as $0.85 < R^2 < 0.95$.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 characterization of Titanium based binary alloys.

## 6. CONCLUSION

Many researchers have worked on monoelements, copper(cu) and silver(Ag) materials to function as contraceptive device, 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. Ti17%Cu alloy, which affects the microstructure and strengthening of mechanical properties of the contraceptive biomaterial (Ti17%Cu), .

### CONTRIBUTION TO KNOWLEDGE

This experimental research has provided a knowledge interface in the development of a beta (β) phase biomaterial alloy(Ti17%Cu)with Bcc microstructure, that are anchored on software designed parametric model equations, thus proferring an improved alternative solution to intractable intrauterine contraceptive challenges of the existing monoelement, copper IUDT380 biomaterial.

### Recommendations For Future Research work

**(1)** Future research will be directed towards employing additional invivo tests on Ti17%Cu ,in animal and human systems, in order to augment the existing in vitro research analysis of Ti17%Cu alloy, with further pursuance of confirmatory approval of NAFDAC for patent certification of this research project.

**(2)** Also, ageing heat treatment process that could optimize the mechanical strength, biocompatibility, Corrosion resistance and antiseptic microbial responses of the Ti17% Cu alloy is recommended.

### ACKNOWLEDGEMENT

The authors wish to acknowledge the contributions and support of the World Bank African Centre of Excellence for sustainable Power and Energy Development, University of Nigeria Nsukka.

## REFERENCES

[1] Ahti, Kosonen (1978), Fertility and Sterility. American Fertility Society

[2] Ali A. L.S (2004), Biomedical Materials Engineering 15, 307

[3] Amir, M. K., Moshe, G., Egan, H., and Doevan, G. L. F. (2015), Properties of Titanium fabrication in Biomedical Applications. *Journal of Biomaterials and Tissue Engineering*.

[4] Chen,C., Feng, X,. and Shin, Y. (2018), Microstructure And Mechanical Properties Of Ti-Cu Amorphous Coatings .Synthezised For Pure CU Sustained By Mechanical Alloying Method HTTP: Dec org 10,007]12598:-018-115-x].

[5] Coatings, D.J. R (ED) (2003), Handbook Of Materials For Medical Devices. Asm In, Material Park Ohio PP 180-192

[6] Conangelo, V. T and Heiser, F. A. (1974), Analysis of Metallurgical failure, willey inter science.

[7] *Erlin Zhang et al (2013).A New Antibacterial Titanium Copper Sintered Alloy,Preparation And Antibacterial Property Of Material Science And Engineering C:33 (4281-4289).*

[8] Gilbert, J. I, Burkley, J. T and Jacobs, .J. (1993), Biomedical Materials Res, 27, 1833.

[9] *Gonzalez E. G, Mirza Rosca J.C (2004). Study of Corrosion Behaviour of Titanium and some of its Alloys for Biomedical and Dental Implant application. Electrochemical Chem.*

[10] *Hong LT., Koo . Cho (2005) . Antibacterial properties, corrosion resistance and mechanical properties of Cu-Modified SS304 stainless steel . Mater . sci. Eng. A(393) ;213 -222.*

[11] *Jin L .G , Liu E. ,Yu F. , Shin X. , Wang H. , (2015) , Microstructure , Corrosion , Tribological And Antibacterial Properties OF TI- CU Coated Stainless Steel .Journal OF Mech .Behaviour. Biomaterial 50; 23-32*

[12] Kalpana, G., Sharon, L., Hillier, T., Howton, M., Pacita, L. R. And Walter, E S. (2009) ,Effects OF Contraceptive method on Vaginal Microbial Flora. A Protective Evaluation.The Journal of Infectious Disease Society, USA, 181:596-600

[13] Paul, D. E., Temple, B. J., and Ronald, K., (1988) Materials and Processing in Manufacturing.

[14] Qzhi, C. and George, A T. (2015). Metallic implant materials. Material science and engineering 287:1-57.

[15] *Sammons R.L (2011).Modifying biomaterial surfaces to optimize interactions with Tissue.Williams (ed).surface modification of biomaterials methods.Analysis and applications.wood head publishing, oxford pp 366-389.*

[16] Sutter, T. and Bonni, (2005), Analytical methods in corrosion science and engineering D. marrevs and F. Manifield editors p. 649, CRC press, New York.

[17] Sykaras, N., Jacopino, A.M, Marker, V.A ,Triplett, R.G ,and Woody, R.D. (2000), Implant materials, Designs and surface topography. Their effect on osseointegration. A literature review .int, journal of oral m,axillo facial implants 15;675 -700.

[18] Udeh,O.U.,Edelugo S.O., (2021),Development and Analysis of an Improved Intra Uterine Contraceptive Alloy for Gynaecological Application(Invitro) ESUT Nigeria.

[19] Udeh,O.U., Edelugo S.O., (2021),Phase/Microstructure effects on the Development and Analysis of an Improved Intra Uterine Contraceptive Alloy for Gynaecological Application, Caritas Nigeria.

[20] Ullman,G. and Hammerstein, J. (1973),Inhibition of sperm mortility in vitro by copper wire contraception. Eleseiver worth Holland Biomedical press, p.243.

[21] *William D.F (1990), Current perspectives on implantable devices. India*

[22] Zielseke, F., Koch, U.J, Bachira, B, and Landenburg, H.(1974), Studies on copper release from copperT-devices (T-Cu200)and its influence on sperm migration,ro. Contraception 10:651-1974.