Evaluation of Tensile Strength of Heat Treated AISI 304 Stainless Steel with Automated Ball Indentation (ABI) Technique

A. U. Iwuoha
Department of Mechanical Engineering
Imo State University, Owerri. Imo State. Nigeria.
Email: nmatoha@yahoo.com.

ABSTRACT: *This paper evaluated the tensile strength and hardness of AISI 304 stainless steel heat treated to temperatures of 400°C, 600°C and 800°C and soaking durations of 30 minutes, 2 hours, 12 hours, 24 hours and 72 hours in each temperature level. The tensile strength was measured using Automated Ball Indentation (ABI) technique while the hardness was on the Brinell scale. To establish confidence in the ABI technique, values obtained were compared with similar studies conducted with the conventional tensile measurement tool. The tensile and hardness properties of type 304 SS increased with treatment temperatures of 400°C and 600°C for all the holding durations up to 72 hours. The long stay was akin to work hardening which increased the tensile strength. The cooling in hot sand bed may have equally contributed to increased surface hardness measured. At temperature of 600°C, precipitation of chromium carbides along grain boundaries coupled with a dense formation of sigma phase and other phases like the chi combined to influence the tensile strength. Temperature of 800°C and soaking duration beyond 30 minutes produced decreased tensile and hardness properties in the steel. At this temperature, transformation of the steel grains by re-crystallisation and relief of internal stresses occurred. The extended soaking duration of 72 hours led to increase of Cr diffusion to the surface.*

Keywords: Tensile Strength, Hardness, Heat Treatment, Type 304 SS, ABI Technique.
Received for publication on 10 April 2013 and accepted in final form 2 May 2013

INTRODUCTION

Austenitic Stainless Steel (ASS) is the widest in use in the industry of all the Stainless Steel (SS) groups. This is due to its combination of strong corrosion resistance qualities in normal and severe corrosive environments with excellent mechanical properties (high tensile strength, good impact resistance and wear resistance). ASS finds application in the nuclear, petrochemical, petroleum, food processing, wood and pulp industries. Other extensive applications include in low and high pressure steam boilers (headers and vessels), fossil fuel fired power plants, flue gas desulphurization equipment, super heater reheating tubing (Higgins, 1994). Domestically, it is the material of choice in the kitchen. In Nigeria, it is extensively employed in Natural Gas Liquids (NGLs) and LNG plants.

ASS is largely highly weldable, the lower alloys being more weldable than the higher alloy variants. The higher the carbon content, the harder the SS and so the more difficult it is to weld. Welding of these stainless steels usually results in inhomogeneous and dendritic microstructures, with the leftover of small amount of delta-ferrite (b.c.c. delta iron) and segregation of major and minor alloying elements at the phase interfaces (Parmar, 2005). The problem of Cr carbide ($Cr_{23}C_6$) precipitation at the grain boundaries during welding and hot cracking of weld bead are common issues. There may also be precipitation of the brittle sigma Fe-Cr phase in their microstructure if they are exposed to high temperatures for a certain length of time as experienced during welding. 700 - 850^{o}C is the temperature range where the transformation from ferrite to sigma or directly from austenite to sigma takes place the fastest (Brooks and Thompson, 2004).

These transformation seriously effect the mechanical and corrosion properties of material. Compared with ferritic and martensitic SS, AISI 304 has superior high temperature strength and reliability. The most common intermetallic phase found during heating austenitic SS is sigma phase which has tetragonal crystalline structure and this is responsible for reduction in toughness at room temperature (Ibrahim, Ibrahim and Khalifa, 2010).

Heat treatment of these steels to obtain certain desirable mechanical and chemical properties is common. The heat treatment may be of quenching, tempering or consists of annealing (to fully austenitize the microstructure, dissolve the carbides and followed by cooling). Depending on the composition and processing of the ASS, the microstructure matrix is that of austenite, undissolved carbides, nitrides, reprecipitated carbides and delta ferrite. It is generally known that properties obtained in these steels are influenced by such heat treatments, the microstructure changes and these exert influence on the properties of these materials such as hardness, tensile strength, toughness, corrosion and wear (Asadollah, Hassan and Ali, 2007). Some previous studies have indicated that inappropriate heat treatments have led to inadvertent microstructural changes (some have produced martensites in an austenite matrix) and hence weakening of mechanical properties of the ASS. This being the case, one of the criteria then, for suitable alloy design, is suitable heat treatment (Davies and Oelmann, 2003).
The hardness and strength levels will depend of soaking time and cooling rate employed during heat treatment. The recommended austenitizing temperatures by various international standards based on composition and heat treatments lies between 950^{o}C and 1100^{o}C. However, these recommendations do not appear to be supported by systematic empirical results (obtained from laboratory experimental studies) correlating various properties to microstructure (Liu Ning, Deng Zhonggang and Huang Menggen, 1991). This work focuses on evaluation of tensile strength of AISI 304 SS in relation to temperature and soaking time using Automated Ball Indentation technique.

AUTOMATED BALL INDENTATION MACHINE (ABI)

This machine is in the group of devices used in metallurgy for in-situ positive material identification (PMI). It is a “non-destructive testing” device, simple and rapid with results. Little material preparation (“specimen preparation”) is needed before testing and no part of the bulk material is removed. The machine makes or leaves a shallow, spherical and unique impression (less than 0.3 mm in depth) in the specimen tested. The material so tested, if found satisfactory, may continue in service without further dressing because no sharp edges as points of stress build-up are introduced (Haggag, Wong, Alexander and Nanstad, 1989). Test results from various investigators (for AISI 304, 316, 310, etc heat treated at different temperatures including long soaking time at 800^{o}C) show that tensile strength levels obtained with ABI machine are in good agreement or reasonably acceptable compared with those obtained from conventional tensile test machines (Haggag, Wong, Alexander and Nanstad, 1989; Murty and Mathew, 1999).

EXPERIMENTAL PROCEDURE

ASS type 304 in plate shape was commercially obtained. Table 1 shows the chemical composition confirmation analysis of the SS. Thickness is 7.75 mm.

Table 1: Chemical Composition of AISI 304 Stainless Steel Material

Element	AISI 304 Stainless Steel Material (% by weight)
Carbon	0.08
Chromium	19.00
Nickel	11.00
Manganese	1.50
Silicon	1.45
Molybdenum	0.00
Phosphorus	0.03
Sulphur	0.002
Iron	Remainder

Heat treatment specimens measuring 70 x 50 x 7.75 mm were cut from the plates Total of 45 pieces. Temperatures of 400°C, 600°C and 800°C were selected for the heat treatment and soaking (holding) duration of 30 minutes, 2 hours, 12 hours, 24 hours and 72 hours were chosen. The specimens were then passed through the heat treatment process on the basis of 3 specimens per temperature per soaking duration. Cooling of specimens was in hot sand bed. Nitrogen gas was used as the protective environment in the furnace.

At the end of process, ABI tensile strength determination specimens were cut from the heated quantities per group. 3 specimens were also prepared from the un-heat-treated (as-received) type 304 plates. Heat treated specimens plus as-received specimens totaled 48. Dimensions of each specimen were 20 x 40 x 4 mm as per machine specification. For each temperature and time, 3 specimens were tested on the ABI device and average taken. Standard deviation was calculated as per the formula stated below:

$$S = \sqrt{\frac{\sum_{i=1}^{n=3}(x_i - \bar{x})^2}{n-1}}$$

Where;

S = Standard Deviation
I = Specimen Number (1, 2, 3)
n = Total Number of Specimens per Group = 3
x_i = Value of Tensile Strength of Specimen
$\bar{x}$ = Average Value of Tensile Strength = $(x_1 + x_2 + x_3)/3$

OR

$$\bar{X} = \frac{\sum xi}{n}$$

The specimens processed through heating were subjected to metallographic examinations for microstructure identification. Before the examinations, specimens were etched following *ASTM-E 3 (2006), Standard test method for metallographic of materials.* Etchant used was saturated solution of ferric chloride ($FeCl_3$) in hydrochloric acid (HCl) with little addition of nitric acid (HNO_3). Etching time was 60 seconds.

RESULTS AND DISCUSSION

Considering the temperature of 400°C, from Table 2, it is seen that the tensile strength of type 304 SS increased with soaking time at that heat treatment temperature; i.e. from 565 MPa for 30 minutes to 620 MPa for 72 hours. This is graphically shown in fig. 1. The Brinell hardness number (BHN) equally increased from 148 (30 minutes) to 165 (72 hours) for the indicated holding times respectively. It is a widely held opinion that longer soaking duration will weaken the mechanically properties (Rajasekhar, Reddy, Mohandas and Murti, 2008), but this is not the case for the 400^{o}C treatment temperature and it could be reasoned that the temperature was low enough to maintain the b.c.c. (magnetic) structure, no re-crystallization took place and the long stay was akin to work hardening which increased the tensile strength. The cooling in hot sand bed may have equally contributed to increased surface hardness measured (Kozuh, Gojic, and Kosec, 2007). Fig. 4 is a graphical display of the hardness trend.

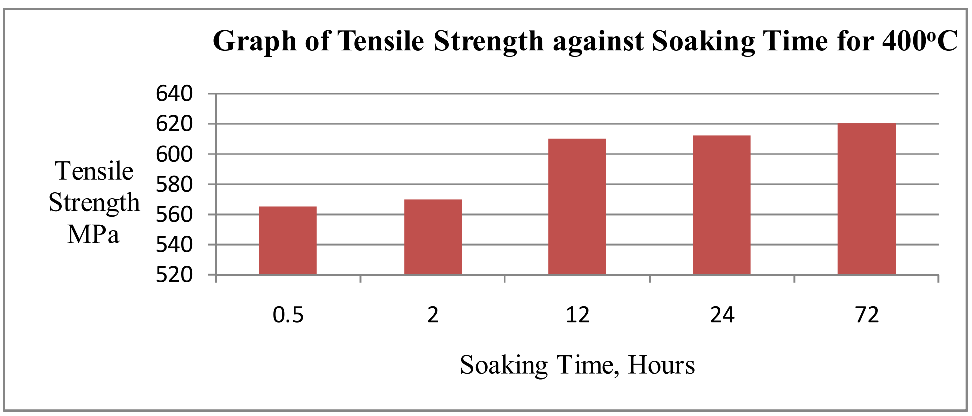
Still referenced Table 2, for 600^{o}C holding time, the same pattern of increase in tensile strength and hardness is followed with increased holding duration in the temperature level. It is noted that 600^{o}C is almost at the threshold of re-crystallization temperature and the durations of 24 and 72 hours may, somewhat, have achieved this. The b.c.c. structure is still maintained since the critical temperature (upper critical temperature) for change to f.c.c. structure was not attained. Type 304 being hypoeutectoid steel, the temperature still fell below the full annealing temperature i.e. about 30 to 50^{o}C above the upper critical temperature. Figs. 2 and 5 graphically show the trends for tensile and hardness properties evaluated. In this temperature, precipitation of chromium carbides along the grain boundaries takes place, a dense formation of sigma phase and other phases like the chi usually occur; all combine to influence the tensile strength (Liu Ning, Deng Zhonggang and Huang Menggen, 1991; Song and Baker, 1996).

Temperature 800^{o}C initially indicated increased tensile and hardness properties for 30 minutes holding duration i.e. from 558 MPa and 148 HB for as-received (un-heat-treated) steel to 605 MPa and 160 HB respectively. Beyond the 30 minutes soaking time, progressively both properties decreased to 560 MPa and 146 HB as the soaking duration increased to 72 hours –an inverse relationship. Figs. 3 and 6 vividly display the decreasing trends for tensile and hardness values. F.c.c. phase was not achieved (b.c.c. was still maintained), 800^{o}C being below the required temperature for that. However, the temperature falls within the process or re-crystallization annealing value for various changes to take place in the steel. The treatment transformed the steel grains by re-crystallisation and relieved internal stresses. Extended holding duration at high temperatures lead to increased chromium diffusion to the surface (Kim and Chang, 2008).

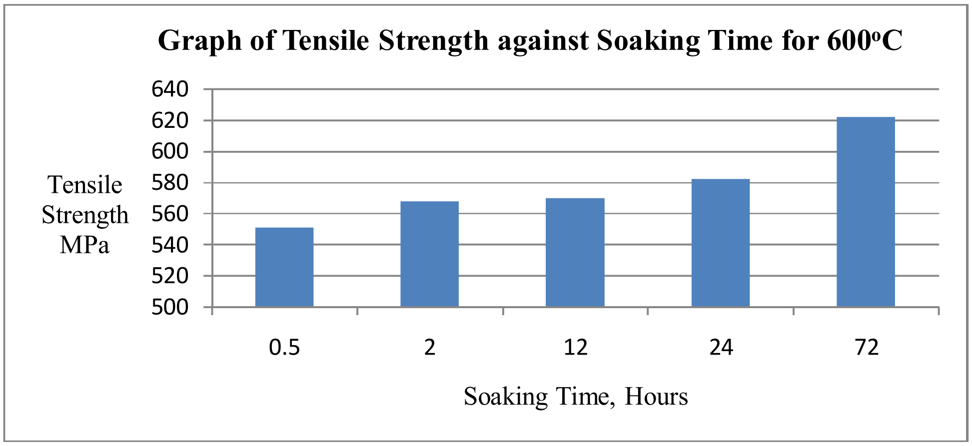
Other researchers (Asadollah, Hassan and Ali, 2007), using conventional tensile testing machine, also found that at the aging temperature of 600^{o}C the delta ferrite was replaced by a maze of brittle sigma phase particles which causes increase in tensile strength of austenitic stainless steel. Smith and Farrar, (2006) found that increasing aging time of austenitic stainless steels at 800^{o}C reduces the impact toughness.

Table 2: Tensile Strength (ABI Technique) and Hardness Values of Heat Treated SS 304

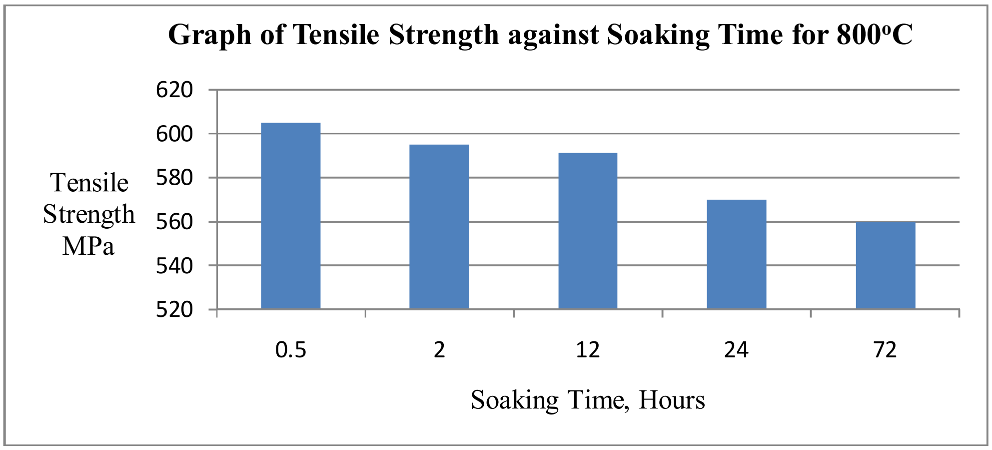
Temperature ^{o}C	Time Hours	Average Tensile Strength (3 Specimens) MPa	Average Hardness (3 Specimens) HB	Standard Deviation ± (Tensile Strength)
As-Received		558	148	3.50
400	0.5	565	148	3.35
400	2	570	150	3.00
400	12	610	160	3.26
400	24	612	162	2.85
400	72	620	165	3.00
600	0.5	551	146	2.98
600	2	568	150	3.26
600	12	570	150	2.65
600	24	582	154	3.15
600	72	622	164	4.00
800	0.5	605	160	3.45
800	2	595	157	2.98
800	12	591	157	3.28
800	24	570	149	3.70
800	72	560	146	3.14



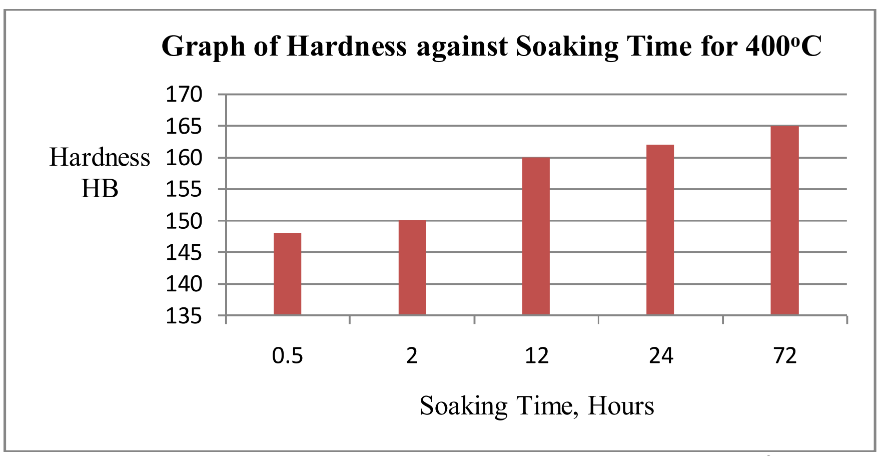
Fig, 1: Graph of Tensile Strength against Soaking Time for 400^{o}C



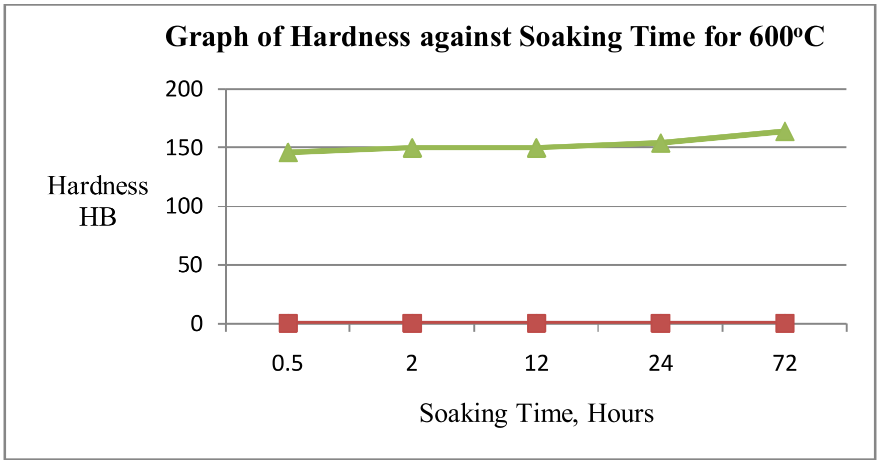
Fig, 2: Graph of Tensile Strength against Soaking Time for 600^{o}C



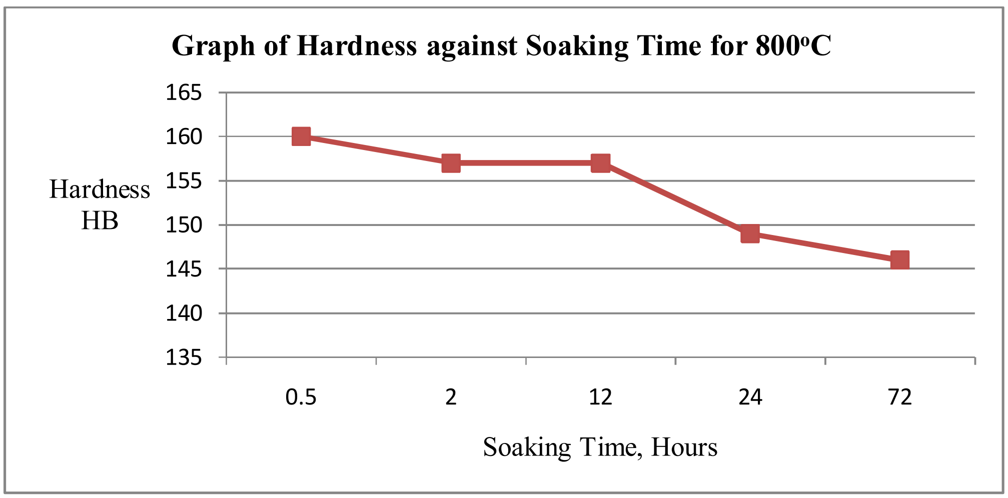
Fig, 3: Graph of Tensile Strength against Soaking Time for 800^{o}C



Fig, 4: Graph of Hardness against Soaking Time for 400^{o}C



Fig, 5: Graph of Hardness against Soaking Time for 600°C



Fig, 6: Graph of Hardness against Soaking Time for 800°C

CONCLUSIONS

The tensile and hardness properties of type 304 SS increased with treatment temperatures of 400°C and 600°C for holding durations up to 72 hours. The long stay was akin to work hardening which increased the tensile strength. The cooling in hot sand bed may have equally contributed to increased surface hardness measured. At temperature of 600°C, precipitation of chromium carbides along grain boundaries coupled with a dense formation of sigma phase and other phases like the chi combined to influence the tensile strength.

Treatment temperature of 800°C and soaking duration beyond 30 minutes produced decreased tensile and hardness properties in type 304 SS. The temperature falls within the process or re-crystallization annealing value for various changes to take place in the steel (transformation of the steel grains by re-crystallization, relief of internal stresses and increase of Cr diffusion to the surface for extended soaking duration).

The tensile strength values obtained by ABI technique are in good agreement or reasonably acceptable compared with those obtained from conventional tensile test machines.

ACKNOWLEDGMENTS
Acknowledgment is due to Engr. Dr. P. N. Atanmo of Anambra State University for discussions and guidance on this work. Acknowledgment is also due to the Engineering team of Standards Organization of Nigeria, Enugu Engineering Laboratory for their assistance during the studies. Finally, special thanks to Saibossam Welds for use of their equipment.

REFERENCES

Asadollah, K., Hassan, F. and Ali, A. A., (2007), Investigation of the effect of aging on the room and high temperature tensile properties and fracture strength of stainless steel 316L weld metal. Proceedings of 8th international fracture conference, 7-9 November, Istanbul, Turkey.

ASM, (1994), ASM Speciality Handbook - Stainless Steels, ASM, Ohio. ISBN 0-87170-503-6.

ASTM E 3 (2006), Standard test method for metallographic of materials, Annual book of ASTM standards, Vol. 03, 0.02, Philadelphia, PA.

AWS B4.0, (2009), Standard Methods for Mechanical Testing of Welds, Miami, Florida.

Brooks, A. and Thompson, A. W., (2004), Microstructural Development and Solidification Cracking Susceptibility of Austenitic Stainless Steel Welds, International Materials Reviews, Vol.79, No.1, 16 – 44.

Chen, X. H., Lu, J. and Lu, K., (2005), Tensile properties of nano-crystalline 316L austenitic stainless steel, Scripta Materialia, Vol. 52, PP. 1039-1044.

Cui, Y., Lundin, C. D. and Vasudevan, H., (2006), Mechanical behavior of austenitic stainless steel weld metals with microfissures, Journal of Materials Processing Technology Vol. 171 150-155.

Davies, D. J. and Oelmann, L. A., (2003), The Structure, Properties and Heat Treatment of Metals, Pitman Books Ltd., London.

Gardi, Ramadhan H., (2012), Tensile strength determination of heat treated austenitic stainless steel AISI 316L using (ABI) method, Al-Rafidain Engineering, Vol. 20, No. 2.

Haggag, F. M., Wong, H., Alexander, D. J. and Nanstad, R. K., (1989), The use of field indentation microprobe in measuring mechanical properties of welds, Proceedings of the 2nd international conference on trends in welding research, Gathlinburg, Tennessee, USA. PP 843-849. 14-18 May, 1989.

Higgins, R. A., (1994), Engineering Metallurgical Processes, 2nd Edition, ELBS, London. Pp. 480.

Ibrahim, O. H., Ibrahim, S. I. and Khalifa, T. A. F., (2010), Effect of aging on the toughness of austenitic and duplex stainless steel weldments, Journal of Material Science and Technology ,Vol. 26, No. 9, PP.810-816, China.

Jim, W. K., (2004), Journal of the Korean Nuclear Society Vol. 36, No. 3, PP 237-247, June, 2004.

Kim, T. K. and Chang, S. (2008), Microstructural observation and tensile isotropy of an austenitic ODS steel, Nuclear Engineering and

Technology, Vol. 40, No. 4, PP. 305-310.

Kozuh, S., Gojic, M. and Kosec, L., (2007), The effect of annealing on properties of AISI 304 base and weld metal, RMZ-Materials and Geo-Environment, Vol. 54, No3, PP. 331-334.

Liu Ning, Deng Zhonggang and Huang Menggen, (1991), Effect of heat treatment on microstructure and mechanical properties of martensitic–ferritic stainless steel containing 17%Cr and 2%Ni, Material Science Technology, 1991; 7:1057.

Mils, W., (1997), Fracture toughness of type 304 and 316 stainless steels and their welds, International Material Reviews, Vol. 42, No. 42, PP. 45-82.

Murty, K. L. and Mathew, M. D., (1999), Non-destructive studies on tensile and fracture properties of Molybdenum at low temperatures (148 & 423 K), Journal of Materials Science, Vol. 34, PP. 1498-1503.

Nnuka, E. E., (2000), Effect of Dopants on the Structure and Properties of Aluminum and some of its alloys, JAST, Vol. 4, No.2.

Parmar, R. S., (2005), Welding Processes and Technology, 2nd edition, Khanna Publishers, Delhi.

Rabbe, P. and Heritier, J., (1999), Properties of Austenitic Stainless Steels and their Weld Metals, Influence of Slight Chemistry Variations, ASTM STP 679.

Rajasekhar, A., Reddy, G. M., Mohandas, T. and Murti, V. S. R., (2008), Influence of post-weld heat treatments on microstructure and mechanical properties of AISI 431 martensitic stainless steel friction welds. Material Science Technology, 2008; 24: 201–12.

Shankar, V., Messler Jr., R. W., (2002), Segregation of Phosphorus and Sulphur in Heat- Affected Zone Hot Cracking of Type 308 Stainless Steel. Welding Journal, 78–84.

Smith, J. J. and Farrar, R. A., (2006), Influence of microstructure and composition on mechanical properties of some AISI 300 series weld metals, International Material Reviews, Vol. 67, No. 1, PP 25-51.

Snykess, M., Bogoerts, W., Bruggeman, A., Embrechts, M., Daenner, W. and Dr Steiner, (2002), Fusion engineering design 8, McGraw-Hill Books. New York.

Song, J. and Baker, T. N., (1996), A study of precipitation in as–welded 316L plate using 316/317L weld metal, Materials Science and Engineering, A212, PP. 228-234.

Sourmali, Z. T., (2001), Precipitation in creep resistant austenitic stainless steels, Material Science and Technology, Vol. 17, No. 1, PP. 1-14.

Sunwoo, A. J., Bradley LLL, E. L. and Morris, J. W. Jnr., (1990), Effects of heat affected zone peak temperatures on the microstructure and properties of 2090 Al alloy, Metallurgical Transactions. Vol. 21 A, No. 10, PP. 2795 – 2804.

Tehovnik, Y. F., Arzensek, B. and Arh, B., (2008), Tensile test of stainless steel in temperature range of 800 to

1200^{o}C, Metallurgiya, Vol. 47, No. 2, PP. 75-79.

Reference to this paper should be made as follows: A.U. Iwuoha (2013) Evaluation of Tensile Strength of Heat Treated AISI 304 Stainless Steel with Automated Ball Indentation (ABI) Technique, *J. of Engineering and Applied Sciences, Vol.5, No.1, Pp. 35-44.*

Biographical Note: A.U. Iwuoha holds B.ENG and M. ENG and he is currently a Lecturer with Department of Mechanical Engineering, Imo State University, Owerri, Nigeria.