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Role of Age Hardening Heat Treatment on the Hardness Values of 93.95 Al-5 Zn-1.05 Sn/5 Al_2o_3- Sio_2 Particulate Composite

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Abstract - The effect of age hardening heat treatment on the hardness values of 93.95Al-5Zn-1.05Sn /5Al_2O_3-SiO_2 particulate composite has been studied. The data used for the work was generated at the National Metallurgical Development Centre, Jos Foundry shop, where the composite was developed. The composite was developed using the stir cast method. The result of the work has shown that the composite has a response to age hardening heat treatment with the optimum hardness value occurring at 2 hours of age hardening. A model was developed for the relationship between age hardening time and hardness of the composite. It was tested, and the product moment of correlation and the coefficient of correlation values attested to the degree of correlation existing between the two variables.
Keywords: Age hardening; Heat treatment; Hardness value; Correlation; Particulate Composite

Introduction

Age hardening heat treatment is an important technique for hardening of metals in the metal industries (Ihom *et al*., 2012a). The process is used to enhance the mechanical properties of a wide range of alloys, notably those based on aluminium but also embracing some nickel and other nonferrous alloys, as well as certain steels (Ihom *et al*., 2012a). It is important that the mechanisms that produce this improvement in properties is understood so that the desired properties can be optimized.' There are four important general methods by which the resistance to plastic deformation of a metal crystal may be increased, namely by cold working ,by refining the grain size, by solid solution strengthening and by precipitation hardening, and many modern high-strength alloys depend on the use of one or more of these effects (Cottrel, 1980).

The first of these methods has been known from ancient times, but the latter technique sprang from Alfred Whim's observations and experiments during the years 1906-1909 in Germany. Wilm searched for an aluminium alloy which could be hardened by quenching from an elevated temperature in a similar manner to steel. In one alloy, known later as 'duralumin' this contains 4% copper, 0.5% magnesium, and a small amount of manganese. It was accidentally discovered that the hardness of quenched alloy was relatively low and subsequently increased with time. This discovery of 'age hardening' represents the only new method of hardening alloys since the quenching of steel was discovered in the second millennium BC (Cottrel, 1980). Age hardening has been attributed to decreasing solid solubility of the solute in the solvent at decreasing temperature which normally results in the precipitation of the second phase when the alloy is quenched and aged for a sufficient time. This phenomena is often observed in aluminium alloy matrix composites, particularly the ones with reinforcing agents in the form of a fine "submicroscopic dispersions and elements like copper magnesium, and manganese (Higgins, 1985).

Composites combine the attribute properties of the other classes of materials while avoiding some of their drawbacks, they are light, stiff, and strong, and they can be tough. Metal matrix composites (MMCs) reinforced with ceramics or metallic particles are widely used due to their high specific modulus, strength, hardness, and wear resistance. MMCs are considered as an alternative to monolithic metallic materials or conventional alloys in a number of specialized applications. The majority of such materials are metallic matrices reinforced with high strength, high modulus and often brittle second phase in the form of fiber, particulate, whiskers, embedded in a ductile metal matrix (Khanna, 2008; Ihom *et al*., 2012b)

The basic structural changes or morphological changes on age hardening are brought together by the different stages of disintegration of the saturated solid solution resulting from hardening of the alloy. Since disintegration of the saturated solution is a diffusion controlled process, the degree of disintegration, type of precipitation from the solution, their dispersion, form, and other structural characteristics depends on the nature of the alloy and its chemical composition. Besides the structure of an age-hardening alloy, it also depends on impurities, heating temperature, velocity of cooling on hardening, plastic deformation after hardening, duration of weathering of the hardened alloy at room temperature before artificial hardening and many other factors. The effect of all these factors combined makes a study of the process of age hardened alloys and composites difficult (Ihom, 2012; Ihom *et al*., 2012c; Curran, 1998; Gandhi and Thompson, 1999). Age hardening assists in the distribution of the particles in the matrix and this goes a long way to improve the mechanical properties. In age hardening no phase transformation takes place what happens is precipitation (Ihom *et al*., 2012b; Ihom, 2012; Ihom *et al*., 2012c; Curran, 1998; Gandhi and Thompson, 1999; Aigbodion, 2007). This increases hardness of the material.

The objective of the work is to study the effect of age hardening heat treatment on the hardness of 93.95Al-5Zn-1.05Sn / $5Al_2O_3.SiO_2$ particulate composite. This study will be carried out using simple linear regression model. This is because the variable shall be limited to age hardening time as the independent variable and hardness as the dependent variable (Ihom *et al*., 2009; Ihom *et al*., 2014; Ihom *et al*., 2006; Ihom *et al*., 2011).

Materials and Methods

Materials

The materials used for this work were pure aluminium from electrical cable company in Kaduna, pure zinc (Zn) 99% and tin (Sn) 98.8% from National Metallurgical Development Centre, Jos, alumino-silicate clay and water for quenching obtained from the foundry shop.

The equipment used for the work were those of the foundry shop of NMDC Jos and these included Rockwell hardness tester (Shimadzu D60), melting furnace (KHE1:170), oven (Pickstone), digital weighing balance (Mettler, 1600), and 12kg weighing balance (Rockweight 12kg). Others were laboratory ball mill, nest of sieves and sieve shaker made by George Fischer +, specimen lathe, quenching bath, watch, ladle and mechanical stirrer.

Procedure

The data used for this work was generated at the foundry shop of the NMDC, Jos. The composite was produced using the stir-cast method of particulate composite production, where the matrix alloy was first produced and the particulates added and stirred mechanically at a steady rate but fast enough to ensure uniform and homogeneous mixing. The stirring should however be smooth enough to avoid turbulence and spillage of the molten metal. This was the method adopted in producing the 93.95Al-5Zn-1.05Sn/ $5Al_2O_3.SiO_2$ particulate composite. The stirring was done at 315rpm when 5 wt. % alumino-silicate particles of 20μm were added. The molten composite was quickly transferred into gravity mould, there it solidified. The specimens were removed from the moulds cleaned and machined into test specimens. The test specimens were solution treated at $500^{O}C$ and quenched in warm water at $60^{O}C$. They were removed and dried after which they were age hardened at $150^{O}C$ for 1-6 hrs in the oven. The age hardened test specimens were allowed to cool after removal from the oven. Hardness test was then conducted on the specimens using Rockwell hardness tester.

Results and Discussion

Development of a simple linear regression model

During the work the only variable was the age hardening time which was varied from 1-6 hrs, so the ageing time was the independent variable and the hardness was the dependent variable. The composition of the composite was constant, and so were the ageing temperature and other factors (Table 1).

Table 1. Hardness value variation with age Hardening time of 93.95Al-5Zn-1.05Sn/ $5Al_2O_3.SiO_2$ particulate Composite

S/no	Age hardening Time (hour)	Hardness HRB
1	1	27.00
2	2	31.53
3	3	27.66
4	4	23.33
5	5	25.83
6	6	26.40

The basic two variable models (one dependent and one independent variable) is

$$Y = a + bX \tag{1}$$

Which can be solved using the normal equations thus:

$$an + b\sum X = \sum Y \tag{2}$$

$$a\sum X + b\sum X^2 = \sum XY \tag{3}$$

Where, n, is number of pairs of figures, a and b are constants representing the intercept and the slope. b is called the regression coefficient X and Y are the variables representing the independent and dependent variables.

The product moment coefficient of correlation r is given by

$$r = \frac{n\sum XY - \sum X \sum Y}{\sqrt{n\sum X^2 - (\sum X)^2} \ X \ \sqrt{n\sum Y^2 - (\sum Y)^2}} \tag{4}$$

The separate regressions of the hardness of the composite as Y_H and age hardening time as X are calculated and shown in Table 2.

Table 2. Calculation of the separate regressions with hardness of the composite as Y_H

S/no.	X (Ageing time, hours)	X^2	Y	Y^2	XY
1	1	1	27.00	729.00	27.00
2	2	4	31.53	994.14	63.06
3	3	9	27.66	765.08	82.98
4	4	16	23.33	544.29	93.32
5	5	25	25.83	667.19	129.15
6	6	36	26.40	696.96	158.40
$\sum$	21	91	161.75	4396.66	553.91

For Regression of the hardness of the composite Y_H on the age hardening time X

The developed regression equation for the relationship of age hardening time and hardness of the 93.95 Al-5Zn-1.05Sn/$5Al_2O_3.SiO_2$ particulate composite is

$$Y_H = 24.52 - 0.7X \tag{5}$$

The product moment of coefficient of correlation for this relationship is $r = 0.49$ and $r^2 = 0.24$ (Coefficient of determination). Equation 5 is the developed mathematical model for the relationship between the hardness of the composite and the age hardening time of the composite ideally the model should be able to predict the hardness of the composite if the relationship is linear. Table 3 gives empirical values of the hardness of the composite alongside with the calculated hardness of the composite using equation 5.

Table 3. Empirical hardness values of age hardened composite alongside calculated hardness values using model equation

Age hardening time (h)	Empirical hardness of the composite HRB	Calculated hardness values of the composite HRB
1	27.00	23.82
2	31.53	23.12
3	27.66	22.42
4	23.33	21.72
5	25.83	21.02
6	26.40	20.32

The results of the regression analysis show that the model in equation 5 represent the relationship between the hardness of the composite and the age hardening time of the composite. The relationship has a product moment coefficient of 0.49 and coefficient of determination of 0.24. Table 3 is the comparison of the empirical values of hardness of the composite with the calculated values of hardness using the developed model.

The results of the study of the relationship between the developed composite and the variation in age hardening time of the composite show that as the age hardening time increases, the hardness of the composite decreases. This relationship is given by equation 5 and supported by the product moment coefficient of correlation which is -0.49. The correlation coefficient is moderately strong and negative. This means a negative product moment of correlation which is in opposite direction to any increase in hardness. As the age hardening time increases the hardness of the composite will decrease. The coefficient of determination of the relationship is 0.24 which means that only 24% of the changes in hardness of the composite are influenced by the changes in age hardening time. This been so, how reliable is the model in predicting hardness value change of the composite with age hardening time? It must be admitted that as observed by several authors, (Ihom *et al.*, 2012a; Cottrel, 1980; Higgins, 1985; Khanna, 2008; Ihom *et al.*, 2012b; Ihom, 2012) the changes during age hardening are very complex and depend on several factors like, diffusion, temperature, time, nature of the alloy matrix, reinforcing agent, cooling rate etc.

According to Higgins (1985) the extent of the formation of coherent precipitates at ordinary temperatures is limited so that strength attains a fairly low maximum value in a few days and this process used to be called age-hardening'. At higher temperatures the formation of coherent precipitates proceeds further and so the strength continues to increase. However, a point is reached where the thermal activation is such that tiny non-coherent particles of Θ begin to form in accordance with phase equilibrium at this point strength and hardness begin to fall rapidly. The use of higher temperature above optimum results in formation of non-coherent Θ virtually 'overtaking' that of the formation of coherent Θ′. Likewise over ageing results in formation of non-coherent Θ, this leads to reduction in hardness. In addition to the explanation by Higgins (1985) the alloy contains reinforcing particles, whose position in the alloy after solidification is purely diffusion controlled. This makes the modeling of the relationship between the hardness of the composite and the ageing time very complex that explains why the developed model could not bring up the optimum time for age hardening as reflected in the empirical result (Higgins, 1985; Khanna, 2008; Ihom *et al.*, 2012b; Ihom, 2012; Ihom *et al.*, 2012c).

Table 3 shows the empirical hardness values of the age hardened composite alongside the calculated hardness values using the model equation. The empirical results showed that at 150°C age hardening temperature, the optimum age hardening time for best result is 2hrs which gave a hardness value of 31.53 HRB. The empirical result showed that at 1 hr of age hardening the hardness value was 27 HRB, at 2hrs the hardness increased to 31.53 HRB, at 3hrs the hardness dropped to 27.66HRB, this behavior is typical of age hardening process it is however very complex to be captured correctly by the regression model used in this work. The empirical result agrees with the work of several authors (Ihom *et al.*, 2012a; Cottrel, 1980; Higgins, 1985; Khanna, 2008; Ihom *et al.*, 2012b; Ihom, 2012; Ihom *et al.*, 2012c; Curran, 1998; Gandhi and Thompson, 1999; Aigbodion, 2007). Looking at the calculated values using the developed model, and comparing them with the empirical values; the calculated hardness values did not have the rising-to- optimum and falling pattern that is characteristic of the age hardening process (Ihom *et al.*, 2012a; Cottrel, 1980; Higgins, 1985; Khanna, 2008; Ihom *et al.*, 2012b; Ihom, 2012; Ihom *et al.*, 2012c; Curran, 1998; Gandhi and Thompson, 1999; Aigbodion, 2007) this is an indication that the model cannot correctly predict the hardness value of the composite after age hardening (Ihom *et al.*, 2009; Ihom *et al.*, 2014; Ihom *et al.*, 2006; Ihom *et al.*, 2011).

The strength of the study is however in the result of the product moment of correlation and the coefficient of determination which all inferred the degree of relationship (correlation) between the hardness value of the composite and the variation in age hardening time (Ihom *et al.*, 2009; Ihom *et al.*, 2014; Ihom *et al.*, 2006; Ihom *et al.*, 2011).

Conclusions

The study `effect of age hardening heat treatment on the hardness values of 93.95Al-5Zn-1.05Sn/ $5Al_2O_3.SiO_2$ particulate composite' has been carried out and the following conclusions have been drawn from the study: the composite has a response to age hardening heat treatment. The optimum time for age hardening the composite was 2 hours, gave the highest hardness value of 31.53 HRB. The developed model for the relationship between the hardness value of the composite and the age hardening time of the composite was tested and found to be flawed, and the product moment coefficient of correlation and the coefficient of determination has inferred the relationship existing between the hardness value of the composite and the age hardening time.

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