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Thermal Ageing Effect on Electro-Mechanical Properties of Work Hardened High Conductive Copper Based Material

M Muzibur Rahman[1,*], S Reaz Ahmed[2] and M Salim Kaiser[3]

1Department of Naval Architecture & Marine Engineering, Military Institute of Science and Technology (MIST), Dhaka-1216, Bangladesh
2Department of Mechanical Engineering, Bangladesh University of Engineering and Technology (BUET), Dhaka-1000, Bangladesh
3 Directorate of Advisory, Extension and Research Services, Bangladesh University of Engineering and Technology (BUET), Dhaka-1000, Bangladesh

**Corresponding author/ E-mail: muzib1061@gmail.com*

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ABSTRACT. *High conductive materials may undergo work hardening in the process of manufacturing and utilization as machine parts. Moreover, these materials face various thermal conditions at operational environment. As a consequence, the electro-mechanical properties of these materials get changed, which in turn affect their operational ability as these materials need to maintain high conductivity along with desirable mechanical properties. It gratifies to investigate the effect of thermal ageing on the electro-mechanical properties and microstructure of high conductive copper based material. In this work, the samples are prepared from copper ingot and alloy collected from local market. From the bulk material, long bars are taken, and they are at first homogenized and solution treated, and then they have been work hardened at different level in two conditions i.e., at room temperature and near recrystallization temperature. Thereafter, a series of experiments are carried out to determine the changes in conductivity, micro-hardness, strength, elongation and microstructure of samples as a function of thermal ageing temperature. Most of the mechanical properties after thermal ageing are found to be influenced quite significantly by work hardening.*

Keywords: Thermal Ageing, Work-hardening, Conductivity, Microhardness, Microstructure

1. INTRODUCTION

Thermal ageing has close relation to electrical and mechanical behavior of conductive materials during valuable operational life [1-3]. At the same time they may undergo work hardening through hot and cold rolling in different circumstances, which result in some significant effects on properties too. While mechanical work is applied on the material towards plastic deformation at room temperature, it leads to anisotropy with the opportunity of increasing stiffness or strength with a possibility of decrease in ductility and malleability due to strain hardening [4-6]. In contrast, while the load is applied at higher temperature i.e., above the recrystallization temperature, it achieves the desired behavior by destroying its original cast structure [7,8]. As a result work hardening temperature contributes to transforms the porous cast structure to a wrought structure with finer grains, enhanced ductility and reduced porosity. In such works, impurities are also broken up and distributed throughout the material [9].

Copper is of cubic crystal structure i.e., face centered cubic (FCC) which provides useful ductility and malleability in annealed form due to the ability of atoms to roll over each other into new positions keeping the metallic bond alright [10]. As a result, the work hardening through rolling of copper material introduces defects such as dislocations into the crystalline structure [11]. These defects interfere with further deformation making the copper material harder and stronger [12]. Annealing can normalize that rolled copper atoms with twinning and de-twinning at a higher temperature. The most suitable annealing temperature is considered as about the half of melting temperature in order to create and grow strain free new grains. The new grains may remove dislocations and other defects caused by the deformation, thus leaving the material in its original soft condition, although may not be in its original shape. However, properly annealed copper items can become harder during storage also due to precipitation hardening as an effect of thermal ageing [13-18]. But a comprehensive characterization for thermal ageing of copper based conductive material subjected to work hardening is yet to be done.

As such, the present paper is an attempt to investigate the effect of thermal ageing at various temperatures on the electro-mechanical properties, for instance, electrical conductivity, micro-hardness, strength, elongation, microstructure etc. of work hardened copper based materials through hot and cold rolling.

2. SAMPLE PREPARATION AND MEASUREMENTS

To investigate the effect of thermal ageing of highly conductive materials after work hardening, copper and one of its alloys have been chosen to be the most prosing candidates. The selected copper and its alloy are commercially available and collected as bulk amount from the local market. The chemical compositions of these two materials have been tested using XRF machine and the results found are shown in table 1.

TABLE -1: Chemical composition of sample materials (mass fraction %)

Material	Cu	Zn	P	Si	S	Cr	Mn	Fe	Ni	Zr	Pb
Commercial Copper Material	99.655	-	0.129	0.182	-	-	-	0.034	-	-	-
Commercial Copper Alloy Material	24.85	28.15	8.93	2.25	1.71	1.15	3.26	23.43	2.38	0.52	3.37

Square bars of size 300 x 15 x 15 mm are made from the collected bulk materials through cutting and machining precisely. In order to prepare the samples for work hardening, the bars are homogenized for eight hours at a temperature of 500°C and solution treated for two hours at a temperature of 700°C in an electric induction furnace. Thereafter, the bars are placed into rollers for work hardening in two steps. At the first step, thickness of few samples is reduced by 40% using hot roll at the temperature of 400℃ (little higher than the recrystallization temperature of Cu) and similarly few samples by cold rolled at room temperature. To carry out such 40% reduction of thickness, a total of 30 roll pass have been applied each of which is of 0.2 mm. After the first step, samples have been allowed to cool down to the room temperature (25℃) and then rolled again at room temperature to reduce the thickness by further 40% to make final reduction of thickness by 80%. As a result, there are four types of materials on the basis of work hardening conditions, such as (i) cold worked copper, (ii) cold worked alloy, (iii) hot-cold worked copper, and (iv) hot-cold worked alloy. From each type of materials, 64 samples have been prepared following ASTM test standard as dog bone shape of size 100 x 6 x 3 mm with gauge length of 25 mm for the tensile tests, and 64 samples as square shape of size 15 x 15 x 3 mm for the observation of electrical conductivity, micro-hardness and microstructure at every corresponding thermal ageing temperature.

Then the samples have been placed in the furnace to have artificial thermal ageing isochronally at various temperatures such as 25 ℃, 100 ℃, 150 ℃, 200 ℃, 250 ℃, 300 ℃, 350 ℃, 400 ℃ and 450 ℃ etc. for a period of one hour. In all corresponding temperature, the electrical conductivity of these thermally aged samples have been measured using an eddy current type digital conductivity meter with an accuracy of ±0.1% IACS (International Annealed Copper Standard), and their micro-hardness values have been examined using Micro Vickers Hardness Tester (HV-100) with the application of 1 kgf load for 10 seconds. The measurements have been repeated at least 10 times for each condition of a sample and the consistent data have been taken for each test to enhance the accuracy level of the result. Microstructures have been observed using optical electronic microscope (OEM) of model BW-S500. The tensile properties of the samples have been investigated using a computer based universal testing machine (Model: SHIMADZU UH-F1000 KNX) with the crosshead speed of 1.5 mm/minute.

3. RESULTS AND DISCUSSION

3.1 Micro-Hardness

The micro-hardness of cold work copper sample is observed as lower than that of hot-cold work, and it has become reverse while ageing is done at 150°C temperature. In this range of the isochronal ageing temperature, the micro-hardness of copper samples depicts very insignificant variation with the average values of about 160 HV for both work hardening conditions. This value is unusually higher than the hardness of copper as element (35-38 HV). While the ageing temperature is increased to 200°C, micro-hardness values are found little higher for both cold and hot-cold work hardening conditions. At the ageing temperature of 200°C, the micro-hardness of cold hardened copper samples becomes 180 HV and that of hot-cold hardened copper samples is 172 HV. The difference in micro-hardness between these two work hardening conditions of copper samples is only 8 HV. Thereafter, the hardness values of both copper samples get reduced with the rising of isochronal ageing temperature to 300°C or higher. As a result, the micro-hardness curves against isochronal ageing temperature show downward trend of for both cold and hot-cold worked conditions of copper samples as depicted by black and red lines respectively in figure 1. However, micro-hardness curve of cold worked copper proceeds below the hot-cold worked samples, though the maximum micro-hardness value is higher cold work than that of hot-cold work.

On the other hand, alloy samples show micro-hardness values of 135 HV and 95 HV for cold work and hot-cold work conditions respectively with gap of 45 HV, while isochronal ageing is done at room temperature. So, the rolling condition has significant influence on the micro-hardness of copper alloy than that of pure copper. While isochronal ageing is done at elevated temperature, the micro-hardness values of both cold and hot-cold hardened alloy samples are coming closer and while the ageing temperature is increased further, their micro-hardness curves show similar pattern with intersections at number of points on green and blue lines in figure 1.

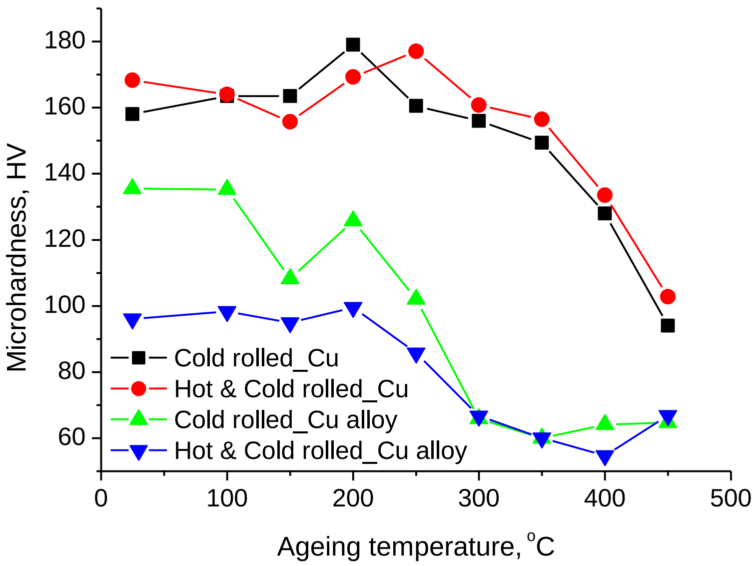

Fig -1: Isochronal ageing curve of micro-hardness of Cu and its alloy, aged for 1 hour.

The isochronal ageing examination has revealed that the maximum hardness, in turn the strength, arises at the ageing temperature of 200°C. As such, isothermal ageing of samples are done at this temperatures i.e., 200°C for both work hardening conditions. The micro-hardness values in this regard are presented in figures 2, which shows that micro-hardness of copper fluctuates at isothermal ageing for 15 minutes and 30 minutes like a transient variation. Thereafter, the results are like steady state condition of showing almost horizontal graphs for all samples for higher period ageing. Another point to note, the hardness values are very closer for both cold worked and hot-cold worked copper. However, the alloy samples indicate significantly different hardness level while work hardening condition is different. The hot-cold worked alloy seems to be very soft with the hardness value of 95 HV in comparison to cold worked alloy with the hardness value of 125 HV after isothermal ageing at 200°C over a period of 4 hours.

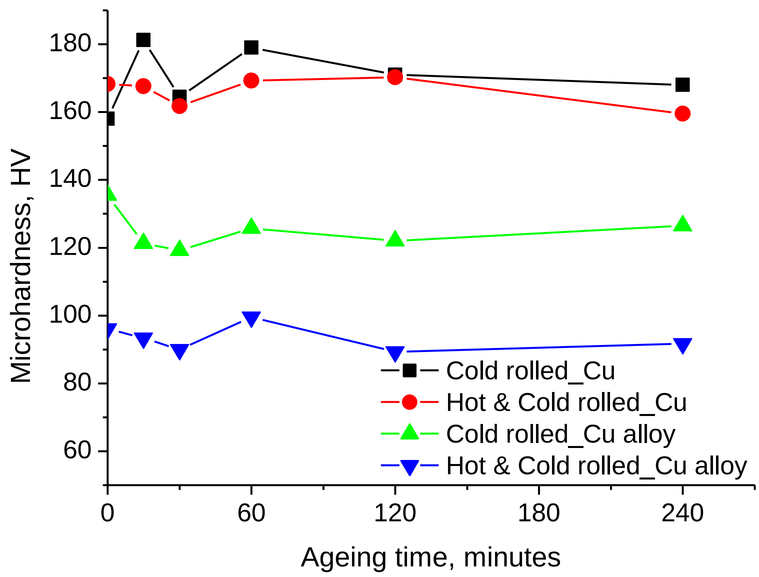

Fig -2: Isothermal ageing effect on hardness of Cu and its alloy as a function of time, aged at 200°C.

3.2 Electrical Conductivity

The electrical conductivity curves of copper samples (black and red lines in figure 3) show the same pattern with a very negligible deviation against the isochronal ageing temperatures for both rolling condition i.e. cold and hot-cold. For the isochronal ageing temperature of 25 to 350 °C, the said both group samples show steady stable values of conductivity with the minimum value of 38 meter/ohm.mm^2. The minimum values are obtained at different ageing temperatures, i.e. at 25°C and 150°C respectively. However, while the ageing temperature is increased to 400°C and higher, the conductivity of copper samples is found accelerated up to 45 meter/ohm.mm^2 at 450°C as shown in figure 3.

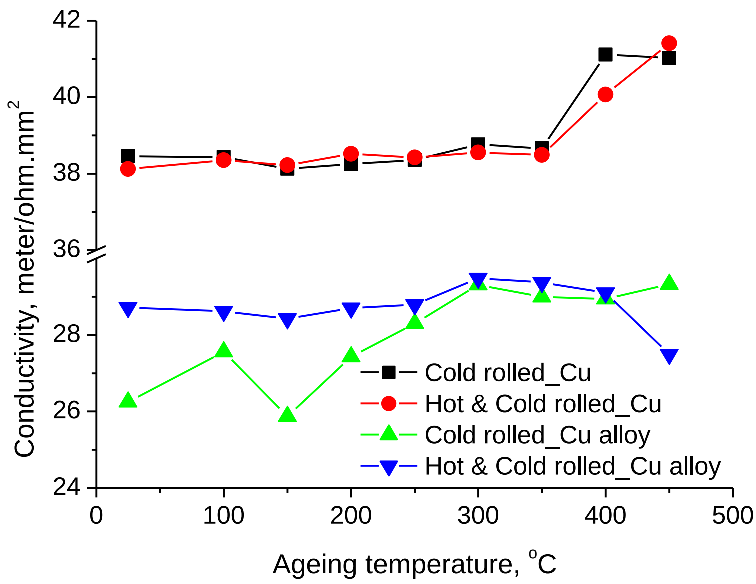

Fig -3: Electrical conductivity variation of Cu and its alloy at isochronal ageing for 1 hour.

The electrical conductivity curves of alloy samples (green and blue lines in figure 3) indicate that the conductivity of the alloy is quite less than that of copper samples for the corresponding thermal ageing temperatures. The conductivity of cold rolled alloy seems here to be varied widely from 25 to 30 meter/ohm.mm^2, whereas the hot-cold rolled alloy shows small variation(28 to 29 meter/ohm.mm^2) over the thermal ageing temperature of 25 to 450 °C.

While isothermal ageing is done at 200°C temperature over a period of 15 to 240 minutes, the conductivity of copper is found unaffected by the rolling conditions, and the value is 38 meter/ohm.mm^2. But the alloy samples show that the conductivity is highly affected by the rolling condition while isothermal ageing is done at 200°C temperature as shown in figure 4. The difference of conductivity in this case is quite high, which gets reduced and remains steady over the time period of 240 minutes with a value of 29 meter/ohm.mm^2 and 27 meter/ohm.mm^2 for hot-cold rolled and cold rolled alloy, respectively.

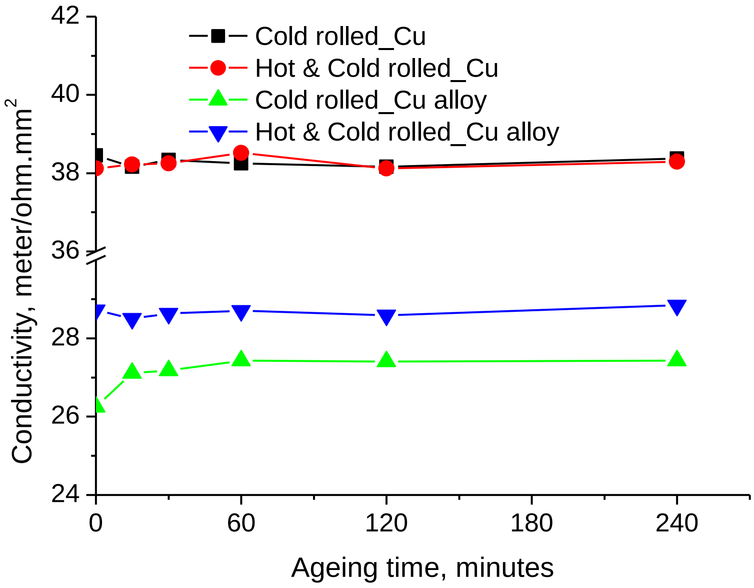

Fig -4: Electrical conductivity variation of cold and hot-cold rolled Cu and its alloy for isothermal ageing, aged at 200°C.

3.3 Ultimate Tensile Strength (UTS) and Elongation

The load deflection characteristics of the sample materials have been investigated through the standard tensile test at cast condition and rolled condition along with accelerated thermal ageing at different temperatures. Here the UTS values of copper and its alloy samples as their cast condition at room temperature have been found as 290.71 MPa and 293.46 MPa respectively. Once they have been cold rolled, the UTS values of copper and its alloy have been increased to 416.65 MPa and 407.49 MPa respectively. Similarly, hot-cold rolled copper has also shown significant increase in UTS value (rise to 442.40 MPa), but the alloy samples have shown insignificant improvement in UTS with the value of 308.15 MPa.

The thermal ageing effect on UTS of cold rolled and hot-cold rolled copper based material is presented in figure 5. The UTS values of both cold rolled and hot-cold rolled copper samples indicate similar increasing variations (black and red lines in figure 5) against the rise of ageing temperature from 25°C to 200°C. Then little fall is observed in UTS values of copper samples while the ageing temperature is 300°C and further again while the ageing temperature is 400°C or above. On the other hand, the alloy samples have shown the falling trends of their UTS with the rise of ageing temperature. At room temperature, the UTS value of cold rolled alloy is 442.395 MPa, which drops to 208.04 MPa at 400°C, thereby reducing strength by 53%. However, the UTS values for hot-cold rolled alloy remain steady up to thermal ageing at 200°C and then it falls at 300°C. The overall UTS patterns against ageing temperatures demonstrate the usefulness of the rolled copper for thermal operating range up to 400°C comfortably, whereas the rolled alloy may be limited to 300°C or even less.

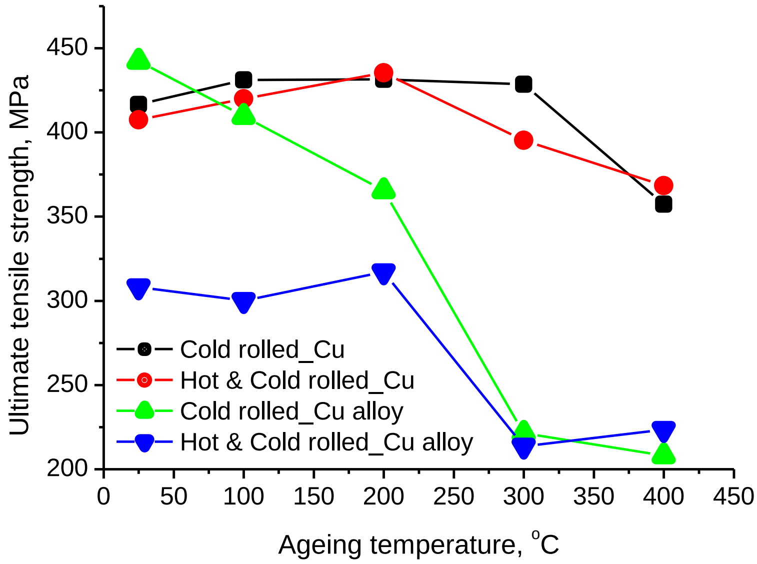

Fig -5: Variation of UTS (10^{-3} s^{-1}) with ageing temperature of cold rolled Cu and its alloy while aged isochronally for 1 hr.

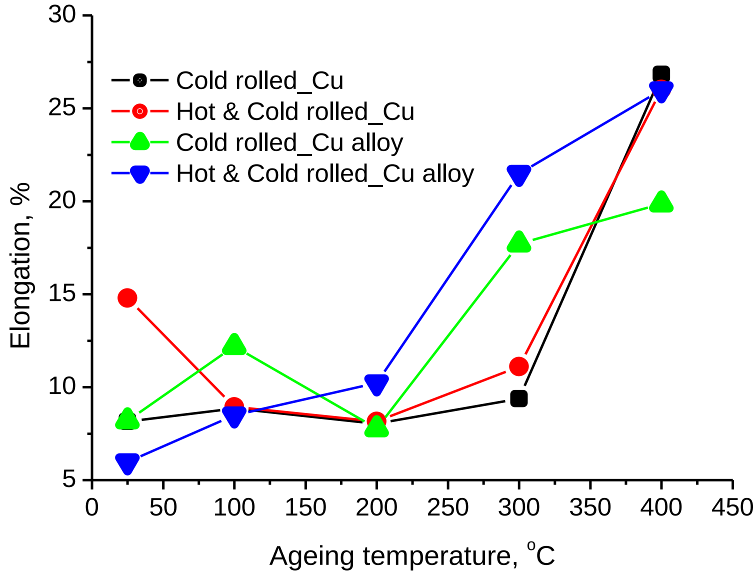

Fig -6: Variation of percent elongation (10^{-3} s^{-1}) with ageing temperature of cold rolled and hot-cold rolled copper and its alloy while aged isochronally for 1 hour.

Figure 6 shows that the ultimate elongations of copper and alloy samples are found significantly affected for both work hardening conditions after thermal ageing. While the rolled copper and its alloy samples have been aged isochronally at 25°C to 200°C, the ultimate elongation have been found steady with less in values, but the percentage of ultimate elongation has been increased while thermal ageing is done at higher temperature like 300 °C and more. This incremental pattern of elongation matches with the reduction rate of UTS.

3.4 Yield Strength and Elastic Modulus

The yield strength values obtained through tensile tests for copper samples as cast, after cold rolled and hot-cold rolled conditions are 192.57 MPa, 352.23 MPa and 282.90 MPa respectively. It indicates that cold rolling has increased the yield strength of copper sample by 83%, whereas hot-cold rolling has increased its yield strength by 47%. Similar behaviour has also been observed for alloy samples. Therefore, the cold work can be considered as a better contributor for improving strength than the hot-cold rolling.

Figure 7 indicates that the yield strength values of cold rolled copper samples remain steady with the rise of ageing temperature up to 100℃. While the ageing temperature is increased further, the yield strength of cold rolled copper increases slowly up to ageing temperature of 300℃ and then fall down. At the same time, hot-cold rolled copper samples show rise of yield strength against the increase of ageing temperature up to 200℃ and then fall down for further increase in ageing temperature 300℃ and more. It indicates that cold rolled copper has higher operational band than that of hot-cold rolled copper. On the other hand, alloy samples have shown almost steady values of yield strength for thermal ageing temperature up to 200℃ and then the strength vs ageing temperature curves show falling down trends. The strength falling trend of alloy sample is gradual and significant, but nonlinear. For the change in thermal ageing temperature from 25℃ to 400℃, the yield strength of cold rolled and hot-cold rolled alloy sample is reduced by 58% and 42% respectively.

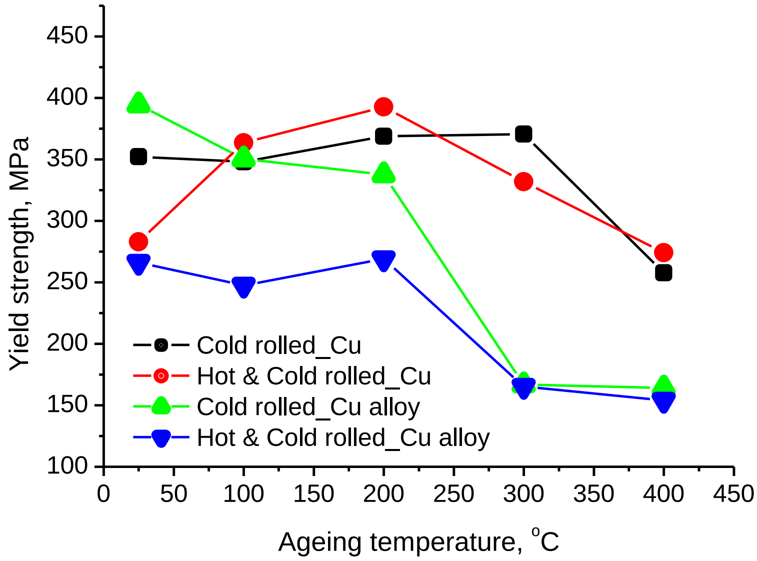

Fig -7: Variation of yield strength (10^{-3} s^{-1}) with ageing temperature of cold rolled and hot-cold rolled copper and its alloy while aged isochronally for 1 hour.

Elastic modulus of copper samples as cast, after cold rolled and hot-cold rolled conditions are 17.06 GPa, 27.13 GPa and 27.13 GPa respectively. It indicates that both cold rolling and hot-cold rolling have increased the modulus value of copper sample by same amount i.e., 59%. On the other hand, Elastic modulus of alloy samples as cast, after cold rolled and hot-cold rolled conditions are 20.66 GPa, 27.36 GPa and 30.87 GPa respectively, which indicates that hot-cold rolling has increased the modulus value 17% more than that of cold rolling for alloy samples.

The elastic modulus variation as a function of thermal ageing temperature for cold rolled and hot-cold rolled copper and its alloy samples is presented in figure 8. While the ageing temperature is increased, the modulus values are reduced and at 400℃ elastic modulus values of both cold and hot-cold copper are reduced by about 45%, and for alloy sample this reduction is about 25%. It indicates the softening of both materials for thermal ageing at elevated temperature is quite significant, and thus the failure mode of these materials is related to the transition from brittle fracture to ductile failure.

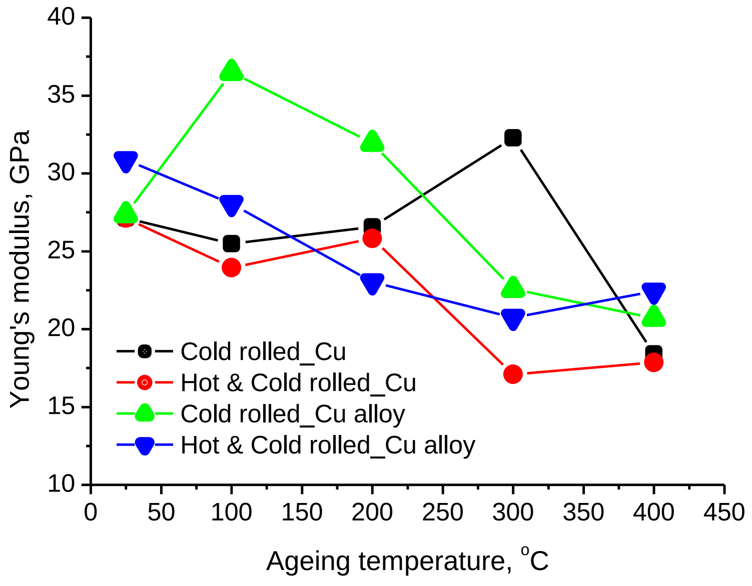

Fig -8: Elastic modulus with the change of ageing temperature for cold rolled and hot-cold rolled copper and its alloy isochronally aged for 1 hour tensile tested at stain rate of 10^{-3} s^{-1}.

3.5 Microstructure

The microstructure of copper samples has a significant geometrical effect on deformation after both cold and hot-cold rolling, and OEM images indicate that grain shapes are changed without changing the grain size. Figure 9 shows one OEM image for each sample after thermal ageing at 400°C. Basically, the thermal ageing at the elevated temperature has changed the grain boundaries with the change in precipitation [11]. When the thermal ageing temperature is done at 400°C, the recrystallization effect makes the change in grains and boundaries. As a result the mechanical properties are influenced against thermal ageing temperature, especially at elevated temperatures.

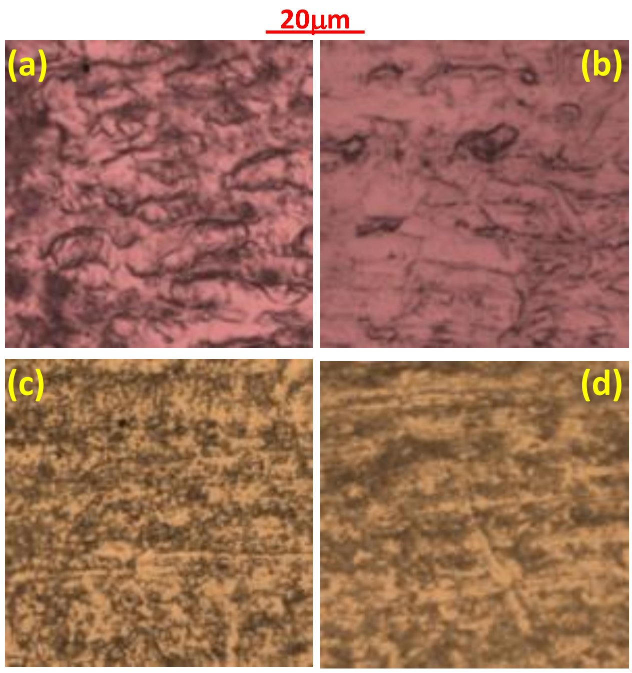

Fig -9: Optical micrographs of (a) cold rolled Copper, (b) hot- cold rolled Copper, (c) cold rolled alloy, and (d) hot-cold rolled alloy after aged at 400°C for one hour

4. CONCLUSIONS

The micro-hardness values of copper and its alloy under cold and hot-cold conditions of rolling are gradually reduced with the rise of isochronal ageing temperature and that at isothermal ageing are found steady. However, the micro-hardness of copper sample is of higher value than that of alloy sample for hot- cold rolling condition. The micro-hardness values are matching with the tensile test results. The result is that the mechanical properties are found to be influenced significantly by the condition of rolling.

The conductivity variation is found insignificantly affected by the rolling conditions, and thus copper wire production through drawing whether at hot or cold condition are safe for maintaining conductivity levels. The micro-structure of both copper and alloy samples indicate significant grain deformation along the rolling pass. But there is no indication to occur any grain refinement after rolling. As a result the increase of strength after rolling can be treated as the outcome of geometrical effect on deformation in the direction of rolling.

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