

AGEING EFFECT ON TRUE STRESS-STRAIN AND FRACTURE BEHAVIOUR OF COLD DEFORMED SOLDER-AFFECTED COPPER

Mohammad Salim Kaiser¹, Shaikh Reaz Ahmed²

¹Innovation Centre, International University of Business Agriculture and Technology
Dhaka-1230, Bangladesh, dkaiser.res@iubat.edu (M.S.K.)
Department of Mechanical Engineering

²Bangladesh University of Engineering and Technology
Dhaka 1000, Bangladesh, srahmed@me.buet.ac.bd (S.R.A.)

Received 09 May 2025

Accepted 25 May 2025

DOI: 10.59957/jctm.v60.i6.2025.23

ABSTRACT

Present manuscript reports the effects of minor Sn - Pb solder on the true stress true strain, impact toughness as well as mode of fracture behaviour of commercially pure copper. Heat treatment of cast alloys is done by homogenizing, solution treatment and quenching those. Then the alloys are cold rolled by 75 % followed by ageing at room temperature, 150°C and 400°C for 1 h each. The results reveal that solder has a positive effect on the tensile properties of the alloy at lower ageing temperature but does not offer enough benefit at higher ageing temperatures. Both Sn and Pb increase strength through solid solution strengthening, with tin performing better than lead because Sn has a different BCC crystal structure and Pb has a similar FCC structure to Cu. In addition, Pb does not form any intermetallic with Cu but Sn forms various intermetallic with Cu as well as impurities which naturally present in the cast alloys. They also have a significant impact on ductility minima of the matrix. The microstructures exhibit relatively thick grain boundaries of minor alloying elements due to the presence of different particles. Fracture surfaces also indicate such particles that inhibit dislocation movement as well as ensure high strength.

***Keywords:** hardness, microstructure, recrystallization, scraped copper, tensile properties.*

INTRODUCTION

With the advancement of engineering science and technology, the requirement of materials in various industrial sectors is increasing. Copper is a widely used metal for electrical components and wires, industrial and marine fields due to its admirable electro-mechanical properties [1]. Since science and technology are developing at a rapid pace and the economy is growing, there is a constant demand for copper and the possibility of using the old/scraped items. It has become important to meet the growing demand considering the degree of copper ore depletion in the natural [2, 3]. However, the scraped copper contains minor solder elements like tin, lead, etc. which causes the speculations amongst the user for the deterioration of required properties.

It should be mentioned that adding elements to an alloy can enhance some characteristics while

having varying effects on others. There is a wealth of information about the impact of alloying elements with copper in a variety of literary works. More precisely, alloyed with Sn decreases conductivity and ductility while increasing strength and precipitation hardening [4]. Although the behaviour is similar, the addition of Al reduces the corrosion properties [5]. While silicon increases machineability and tensile strength, it is less corrosion-resistant than other Cu alloys. Alloys with Ni added exhibit excellent thermal stability, allowing them to maintain their mechanical properties at elevated temperatures. However, their high toughness leads to poor machinability [6, 7]. Again, the trace amount of Zr is used to refine the grain structure and thermal stability, Sc furthermore provided the precipitation strengthening of the alloy [8, 9].

So, it is earnestly important to investigate the behaviour of solder affected Cu and the characterization

of its various properties to nullify the sceptical behaviour and to investigate the possibility of reusing scrap copper to manufacture new components. Materials' fracture failure is a highly intricate micromechanical process. For reasonable accuracy of the calculation results in numerical simulation, the selection of precise material damage models and damage parameters is crucial. In view of the above, few of the properties have been investigated under a research project and interesting findings have been obtained on electro-mechanical, sliding friction and corrosion behaviours of solder affected scraped copper [10 - 12]. It is also necessary to carry out the experimental investigations to address the true stress-strain behaviour of such scraped Cu after thermal ageing to explore the worthy engineering applications. Since lead and tin concentrations in waste copper are typically low, four categories of sample, such as, copper ingots, low tin and lead added copper-tin and copper-lead alloys together with solder affected scraped copper have been taken to carry out investigations. The study mainly deals with the effects of trace amount of soldering in work-hardened samples on true stress-strain and fracture behaviour at the plastic region under tensile loading. Furthermore, the alloys' hardness, impact, and optical microstructural results under various aging circumstances have been documented.

EXPERIMENTAL

Old/scraped copper wires as well as soldered connecting structures after long used were collected from various places. It is melted using a conventional pit furnace that runs on natural gas. It has been noted that the cast sample has minor amounts of Sn and Pb in addition to trace amounts of other elements with copper. Therefore, it has been felt that the effects of Sn and/or Pb inclusion on the properties of Cu are crucial to the investigation of its potential for reuse. To ascertain the distinct effects of Sn and Pb present in old solder-affected

copper, three additional materials were taken: minor Cu - Sn alloy, Cu - Pb alloy, and commercially pure copper. The chemical compositions of all four developed materials were analysed using the spectrochemical method; the results are displayed in Table 1.

The cast samples were machined to create the dimension of $300 \times 15 \times 12$ mm for each type of material. Before machining samples were initially homogenized for time interval 8 h at 500°C , the solution treated for time interval two hours at 700°C and then quenching into ice salt water. Afterward, the plastically deformation was done using a laboratory scale rolling machine under 1.0 mm reduction per pass. As a result, the thickness of the samples became 3.0 mm as of the 12 mm thick. Cold rolled samples were aged at three thermal treatment conditions, i.e., (i) at room temperature (under aged), (ii) at 150°C at which the hardness and ultimate tensile strength and were found to the maximum (peak aged), and (iii) at 400°C (over aged) for the duration of 1 h each [13].

The Instron testing machine was utilized to conduct the tensile test at room temperature, while maintaining a strain rate of 10^{-3} s^{-1} . The samples were prepared in accordance with ASTM specifications to ensure accurate testing. The gauge length of each sample was set at 25 mm. A total of seven tests were performed under each condition, and the average value closest to the true stress-strain curve was selected. The measurement of hardness for various alloys under different annealed conditions was conducted using a Mitutoyo HM-200 Series 810-Micro Vickers Hardness Testing Machine. Both Vickers and Knoop Indenters were utilized with a 100 g load, and an indenter approach speed of $60 \mu\text{m s}^{-1}$ was employed. The test force dwell time was set at 20 sec. For this microhardness measurement, a sample size of $3 \times 15 \times 15$ mm was chosen. At different locations on each polished surface created by the fine grade emery paper of the aged, hardened samples, a minimum of 10 indentations were made. The heat-treated specimens employed were conventional $10 \times 10 \times 55$ mm samples

Table 1. Alloys chemical composition by wt. %.

Alloy	Cu	Sn	Pb	P	Si	Fe
Pure Cu	99.986	0.000	0.000	0.011	0.003	0.000
Cu-Sn	98.456	1.134	0.012	0.270	0.142	0.000
Cu-Pb	98.433	0.002	1.197	0.256	0.113	0.000
Cu-Sn-Pb	97.113	1.257	1.195	0.262	0.173	0.034

featuring a V-shaped notch, 2 mm in depth, positioned at a 45° angle. The assessment of impact resistance was conducted following the guidelines set by ASTM. Each test involved the evaluation of seven test pieces. The optical metallography of alloy specimens was taken in the usual way where Versamet-II microscope was used. In case of using metallographic after being polished with alumina, copper etchant like 1:1 ratio Ammonium Hydroxide and Hydrogen peroxide (3 %) was used. The JEOL scanning electron microscope was utilized to conduct the SEM investigation of the fracture surfaces following tensile failure.

RESULTS AND DISCUSSION

True stress-strain

At room temperature, commercially pure Cu, Cu - Sn alloys doped with tin, Cu - Pb alloys doped with lead and Cu - Sn - Pb alloy affected by solder are subjected to tensile testing. These materials have undergone a 75 % cold deformation and are tested with a strain rate of 10^{-3} s^{-1} . The under-aged, peak-aged, and over-aged conditions as designated in the experimental section, the obtained true stress strain plots are represented in Figs. 1, 2, 3. With out any ageing treatment that is under aged condition, the curves demonstrate some deviation between the slope of the true stress-strain curves and their tensile strength (Fig. 1). Commercially pure Cu display the lowest values of stress, and the solder affected alloy has the highest followed by individually Sn and then Pb added alloys. The reason behind this solid-soluble Sn and Pb in the α -phase alloy, creating a lattice distortion stress field that effectively prevents dislocation movement and thus effectively increases the tensile strength. At the same time, Sn offer higher strength as it forms different intermetallics with Cu namely Cu_3Sn , Cu_6Sn_5 etc. but Pb does not form. However, Sn shows the superior strength because of the dissimilar crystal orientation BCC of Sn precipitated within the FCC Cu matrix whereas FCC Pb precipitates within FCC Cu matrix. β -Sn precipitates might have generated coherency strain which leads to higher strength in this alloy which probably is absent α -Pb precipitates in Cu-Pb alloy. Consequently, the solder affected Cu - Sn - Pb alloy displays the highest strength [14, 15].

When aged at 150°C for one hour, that is the peak aged condition, the slopes of the graph decrease as

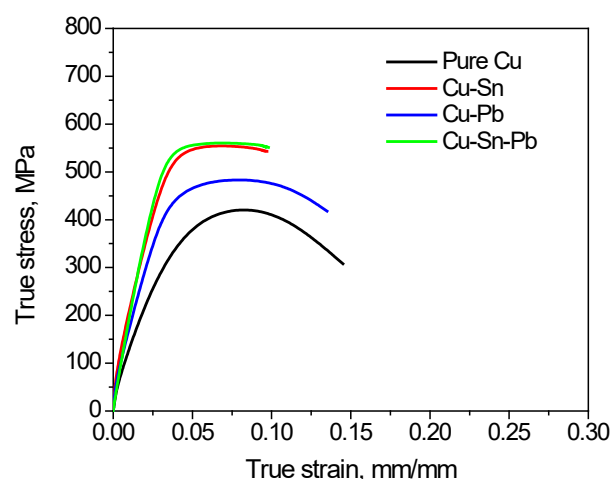


Fig. 1. The experimental alloys' true stress-true strain curves at under-aged condition.

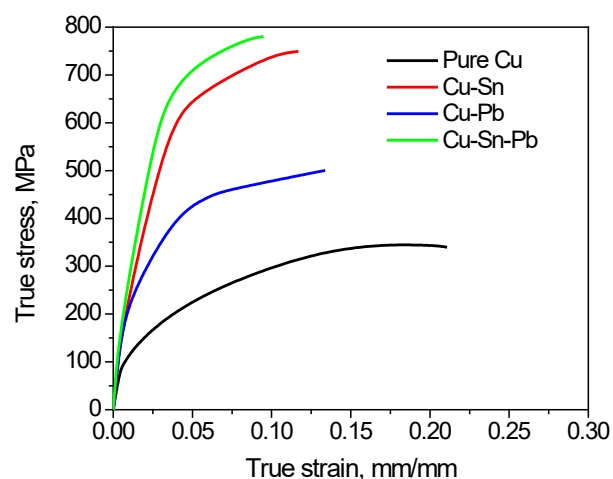


Fig. 2. The experimental alloys' true stress-true strain curves under peak-aged condition.

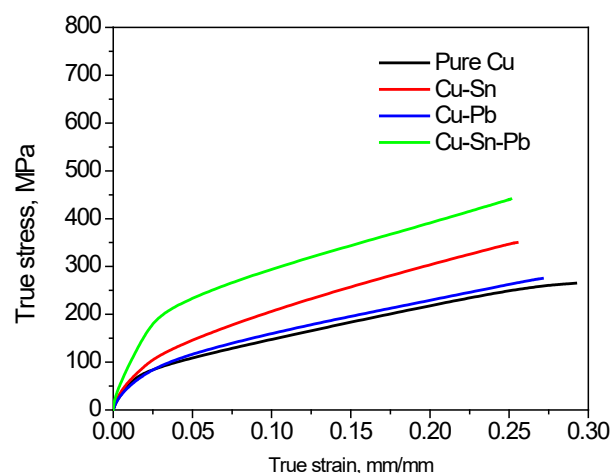


Fig. 3. The experimental alloys' true stress-true strain curves under over-aged condition.

displayed the under-aged condition (Fig. 2). These can be explained through the dislocation theory. It has already been stated that Sn forms intermetallics with Cu. Additionally it forms intermetallics with trace impurities comes from the casting environment and Pb does not form to active in intermetallic formation as Sn. The low number of fine precipitates acts as barrier to dislocation movement resulting in increased tensile strength especially for Sn added alloy. Considering of precipitates Sn added alloy, Pb added alloy and pure Cu follow the increase in tensile strength by solder affected alloys. These precipitation sequences are reflected in the form of ductility minima [13].

When the alloys are aged at 400°C for 1 h, the slope of the stress-strain curves get higher as it reached at over-aged conditions (Fig. 3). This is due to dislocation rearrangement, recovery and precipitation coarsening of the alloys. Dispersed fine precipitates to form coarse precipitates which lose its resistive properties to inhibit dislocation movement [16 - 18]. As a result, higher elongation is observed by all the alloys.

Young's modulus

The result of Young's modulus of the discussed samples under three different ageing condition is presented in Fig. 4. A minor in increasing of the Young's modulus of the elements' added alloys are observed at under aged condition. These differences can be concluded by solute solution strengthening where Sn doped alloys show the higher effect as it form different intermetallics also. At the peak aged condition except the base material Cu, all the alloys are achieved some magnitude. Pure Cu losses its strength due to ageing so some reduction occurs in Young's modulus. For other alloys, some precipitates are forms which hinder the dislocation movement as a result lower elongation and higher tensile strength. So, the proportion of these parameters called the Young's Modulus move to the higher direction. As usual Sn doped alloys display the higher values for the higher level of intermetallic formation. Similarly, at over aged condition the alloys show the lower strength along with higher elongation as a result a drastically decrees in the modulus. High ageing temperature rearranges the dislocations through recovery and recrystallization, and coarse precipitates are not as effective as fine ones in imparting sufficient rigidity to the alloy by inhibiting dislocation movement [13].

Microhardness

The microhardness behaviour of the experimental alloys under the mentioned aging conditions was measured to compare, and the average values are displayed in a bar chart shown in Fig. 5. The hardness outcomes align completely with the tensile behaviour observed during the aging process of the alloys. Typically, minor added alloys exhibit higher hardness values compared to pure copper at the beginning due to the solid solution strengthening of Sn and Pb phases. Higher hardness for Sn added alloy is due to its different crystal structure from Cu as well as Pb has the similar crystal lattice to Cu. The FCC and BCC lattice alloys exhibit different supersaturated solid solution patterns with higher internal stress than that of both similar lattices. In this case cold rolling also initiates to form complex structure. One thing may be noted that atomic size of the Sn is higher than Pb as well as Cu. As a result, the addition of Sn to the Cu matrix accelerates the internal stress. Pb element does not form any intermetallic with Cu, but Sn does. Solder affected

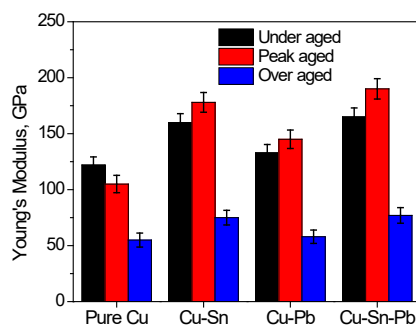


Fig. 4. Variations in the experimental alloys' Young's modulus at them under aged, peak aged and over aged situation.

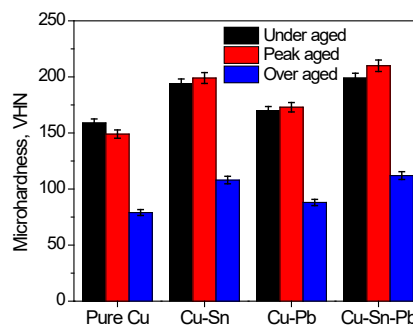


Fig. 5. Variations in the experimental alloys' micro hardness at them under aged, peak aged and over aged situation.

alloys bear the highest hardness for both alloying effects. At the peak aged condition element added alloys show some improvement of hardness. It may be elucidated that ageing treatment help to form different intermetallic with impurities as deposited during casting which contributes significantly to the alloy's strengthening. These intermetallics hinder the dislocation movement under ageing treatment so such type of nature observed [19]. At the over aged condition, all the alloys drastically losses its hardness. Due to extensive coldworking, the strain energy associated with various lattice defects, such as dislocations and vacancies, is significantly increased, resulting in improved hardness. When subjected to higher temperatures during the aging process, several important phenomena occur, including stress relief, dislocation rearrangement, recovery, recrystallization, and grain growth in the alloys. This temperature-induced atomic migration within the crystal lattice leads to a reduction in the number of dislocations, ultimately causing a change in hardness. As recovery progresses, the deformed grains soften and the subgrains rotate and change to a new orientation. It is important to acknowledge that annealing encompasses the process of recrystallization, wherein a blend of cold working and subsequent heating induces the formation of fresh stress-free crystals within a matrix [20, 21].

Impact toughness

Another bar chart as in Fig. 6, provides the parameters of impact toughness for four samples at those three mentioned ageing conditions. Except the base sample Cu, all other minor added samples lose its impact energy due to solid solution strengthening. The impact energy declines at peak aged condition. It can be attributed to formation of the precipitation phases as discussed earlier. These precipitation sequences are reflected in the form of ductility minima. Fine precipitates act as early nucleation sites for micro voids. As a result, there is a decline in the fracture resistance behaviour of the material. The precipitated particles' pinning effect also decreases total elongation. As usual Sn reacts more for its higher intermetallics formation. Again, at the over aged condition the energy reduces more, it may clarify as over ageing condition owing to microstructural change through equax grain like cast alloys as well as precipitation coarsening which stay at grain boundary [22, 23].

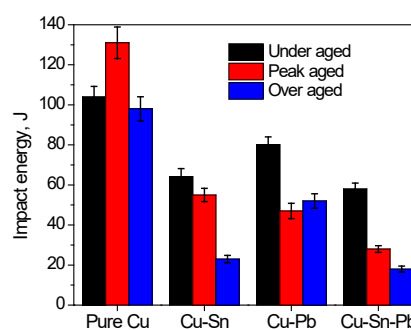


Fig. 6. Variations in the experimental alloys' fracture toughness at them under aged, peak aged and over aged situation.

Optical micrograph

Optical micrographs of solution treated in addition to 75 % plastically deformed discussed alloys are shown in Fig. 7a - d. It is also aged for 60 min at 150°C to reach their peak aged condition. All the microstructure consists of heterogeneous grains by dispersed in rolling directions. Extensive plastic deformation destroys grain boundaries to form subgrains. The minor addition of elements does not provide the foremost differences. But some deviations can be distinguished between alloys as grain boundaries are relatively thin for Cu, followed by Pb doped, Sn doped, and solder affected alloys. Copper materials contain the minimum number of impurities like Fe, C, P, etc., so the intermetallics are smaller in both quantity and size during solution treatment. Tin forms denser grain boundaries than lead because alloys with FCC Cu and BCC Sn lattices exhibit different supersaturated solid solution patterns than similar lattices of both Cu and Pb. Additionally, the atomic size of Sn is larger than that of Pb as well as Cu, so tends to allocate to grain boundaries resulting in denser grain boundaries for alloys containing Sn. Both material effects dominate the dense boundary of the solder affected Cu [15, 24].

SEM analyses

The impact of solder and individually Sn and Pb on the fracture behaviour of pure Cu was explored by SEM analysis as shown in Fig. 8. To determine the fracture surface, cold rolled by 75 % alloy samples were subjected to aging treatment at 150°C for 1 h and concurrently put through to tensile test. The tensile fracture mode of highly deformed Cu is mixed

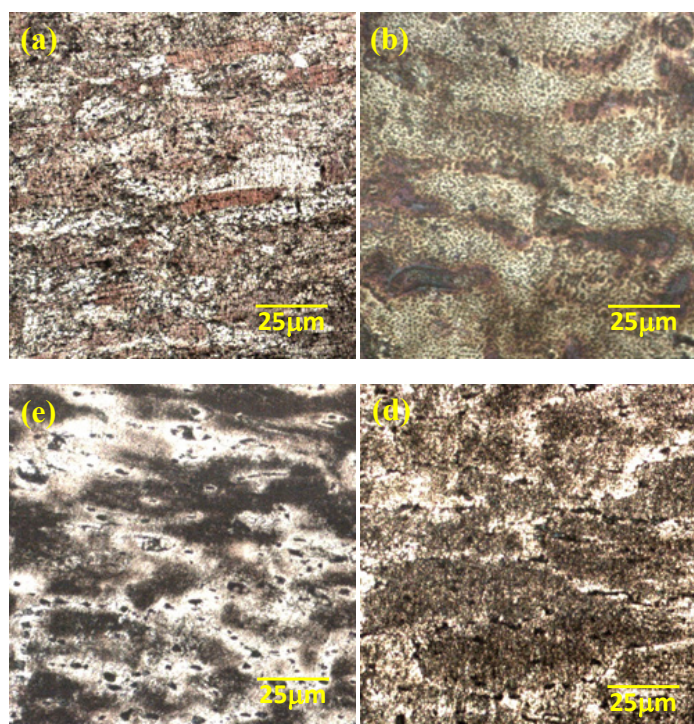


Fig. 7. Optical microstructure of (a) pure Cu, (b) Cu - Sn, (c) Cu - Pb and (d) Cu - Sn - Pb samples with solution-treated that were 75 % cold-rolled and aged for 60 min at 150°C.

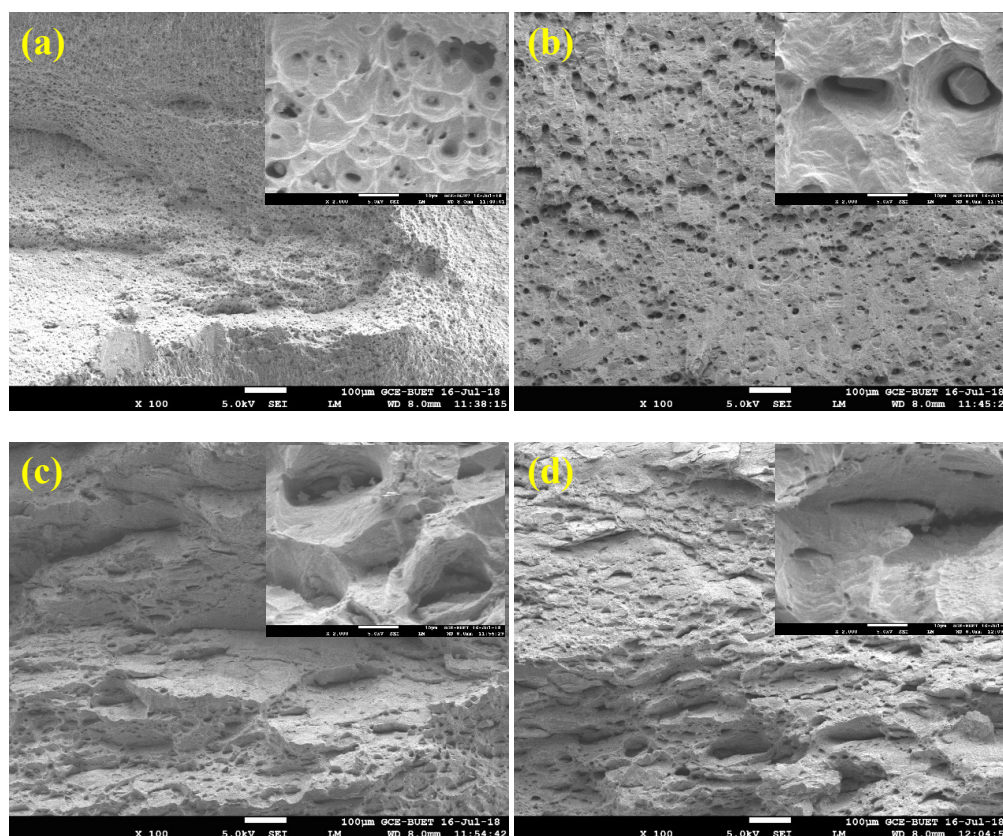


Fig. 8. SEM fractography of (a) pure Cu, (b) Cu - Sn, (c) Cu - Pb and (d) Cu - Sn - Pb samples that aged at 150°C for 60 min and tensile tested at a strain rate of 10^{-3} s^{-1} .

intergranular and trans-granular ductile fracture (Fig. 8a). Trace second-phase particles distributed along grains and grain boundaries promote microcracks and are the main cause of fracture failure. Several small dimples are observed which confirm the typical nature of ductile fracture. Alloys containing minor addition consist of fewer dimples with deep equiaxed volume. Additionally, some smooth and flat areas interspersed with bright ridges are observed. It is because of fracture initiated through thicker grain boundary as created by the elements. Whereas Sn addition demonstrates the deeper dimple for its higher atomic size (Fig. 8b). Since Pb does not form various intermetallic, as well as lower atomic size than Sn resulting low deeper dimples (Fig. 8c). The solder-affected alloy fully mimics the constitutive nature of combining Sn and Pb fracture surfaces (Fig. 8d) [13].

CONCLUSIONS

The following can be drawn as conclusions from the above study of mechanical and fracture behaviour of solder affected copper:

Minor Sn-Pb solder has a great impact on the strength of the commercially pure copper. Solute solution strengthening is responsible for these. Sn shows high strength than that of Pb due to its BCC crystal structure which is different from FCC Cu crystal structure like Pb. In addition, Sn forms different intermetallic with Cu as well as impurities which naturally present in cast alloys but Pb does not form intermetallic with Cu. At higher ageing temperature deteriorates alloys strength due to over ageing effects.

In case of true stress true strain aged alloys display the lower slope as formed precipitate hinders the dislocation movement. At higher ageing temperature the slope rises due to precipitates coarsening through recovery and recrystallization of the alloys.

Minor added alloys losses its impact strength due to solid solution strengthening and reduces more at peak aged condition to form the fine precipitates. These act as the early nucleation site for microvoids resulting decrease in the fracture resistance and ductility minima of the material.

The microstructures of all four samples exhibit elongated grain at the rolling direction and relatively thick grain boundaries of minor alloying elements due

to the presence of different particles. The fracture surface also indicates such particles that inhibit dislocation movement as well as ensure high strength.

Acknowledgements

Thanks to DAERS office and NCE Department of Bangladesh University of Engineering and Technology, Dhaka for providing the laboratory facilities. The corresponding author would like to express his gratitude towards the Treasurer & Director Administration at International University of Business Agriculture and Technology, Dhaka for her appreciated care and encouragement in promoting research activities at the university.

Authors' contributions: M.S.K: Conceptualization, Formal analysis, Investigation, Methodology, Writing - original draft; S.R.A.: Conceptualization, Funding acquisition, Writing - review and editing.

REFERENCES

1. L. Collini, Copper Alloys - Early Applications and Current Performance - Enhancing Processes, InTechJanezaTrdine, 2012, Rijeka, Croatia.
2. J. Cui, E. Forssberg, Mechanical recycling of waste electric and electronic equipment: a review, J. Hazard. Mater., 99, 3, 2003, 243-263.
3. M. Villena, F. Greve, On resource depletion and productivity: The case of the Chilean copper industry, Resour. Policy., 59, 2018, 533-562.
4. C. Zeng, B. Zhang, A.H. Eftefagh, H. Wen, H. Yao, W.J. Meng, S. Guo, Mechanical, thermal, and corrosion properties of Cu-10Sn alloy prepared by laser-powder-bed-fusion additive manufacturing, ADDIT MANUF, 35, 2020, 1-8.
5. A.A. Khan, S. Kaiser, M.S. Kaiser, Electrochemical corrosion performance of copper and uniformly alloyed bronze and brass in 0.1 M NaCl solution, Rev. Mex. Fis., 69, 5, 2023, 1-10.
6. M. Afifeh, S.J. Hosseinipour, R. Jamaati, Manufacturing of pure copper with extraordinary strength-ductility-conductivity balance by cryorolling and annealing, CIRP-JMST, 37, 2022, 623-632.
7. S. Constantinescu, A. Popa, J.R. Groza, I. Bock, New high-temperature copper alloys, J. Mater Eng.

- and Perform., 5, 1996, 695-698.
8. M.S. Haque, S.A.R. Khan, M.S. Kaiser, Effect of Sc and Zr on Precipitation Behaviour of Wrought Al-Bronze. IOP CONF. SER. MATER. SCI. ENG, 1248, 1, 2022, 1-12.
 9. R.F. Tylecote, The Prehistory of Metallurgy in the British Isles: 5, 1st edition, Taylor and Francis, London, UK, 1986.
 10. M.M. Rahman, S.R. Ahmed, Dry Sliding Friction and Wear of SnPb-Solder Affected Copper against Stainless Steel Counter Surface, Ran. Mater. Sci. Eng., 18, 4, 2021, 1-12.
 11. M.M. Rahman, S.R. Ahmed, M.S. Kaiser, Behavior of work hardened SnPb-solder affected copper on corrosion resistance in pH varied environments. EJMSE, 5, 4, 2020, 199-211.
 12. M.M. Rahman, S.R. Ahmed, M.S. Kaiser, On the Investigation of Reuse Potential of SnPb-Solder Affected Copper Subjected to Work-Hardening and Thermal Ageing, Mater. Charact., 172, 2021, 1-19.
 13. M.M. Rahman, S.R. Ahmed, Effects of work-hardening and post thermal-treatment on tensile behaviour of solder-affected copper, Pro. of the Institution of Mechanical Engineers, Part L: Journal of Materials: Design and Applications, 237, 5, 2023, 981-1005.
 14. A. Leineweber, The Cu-Sn System: A Comprehensive Review of the Crystal Structures of its Stable and Metastable Phases, J Phs Eqil and Diff., 44, 2023, 343-393.
 15. J. Peng, J. Li, B. Liu, Q. Fang, P.K. Liaw, Origin of thermal deformation induced crystallization and microstructure formation in additive manufactured FCC, BCC, HCP metals and its alloys, Int. J. Plast., 172, 2024, 1-21.
 16. A. Ahmed, N. Iqbal, M.S. Kaiser, S.R. Ahmed, Thermo-mechanical and optical characteristics of cold-rolled copper with natural melting impurities, AIP Conf. Proc., 2324, 1, 2021, 0300171-7.
 17. J.C. Kim, B.H. Ko, I.H. Moon, On some physical properties of nanostructured Cu-Pb alloy prepared by mechanical alloying, Nanostructured Materials, 7, 8, 1996, 887-903.
 18. P. Yang, D. He, W. Shao, Z. Tan, X. Guo, S. Lu, K. Anton, Study of the microstructure and mechanical properties of Cu-Sn alloys formed by selective laser melting with different Sn contents, J. Mater. Res. Technol., 24, 2023, 5476-5485.
 19. S.L. Semiatin, Metalworking: Bulk Forming, Volume 14A, ASM International, 2005, Almere, Netherlands.
 20. Y. Minggao, W. Qingsui, X. Kuangdi, Recovery and Recrystallization. In: Xu, K. (Eds.) The ECPH Encyclopedia of Mining and Metallurgy, Springer, Singapore, 2023.
 21. D. Raabe, 23 - Recovery and Recrystallization: Phenomena, Physics, Models, Simulation, Physical Metallurgy (Fifth Edition), Elsevier, 2014, 2291-2397.
 22. L. Fan, T. Yang, Y. Zhao, J. Luan, G. Zhou, H. Wang, Z. Jiao, C.T. Liu, Ultrahigh strength and ductility in newly developed materials with coherent nanolamellar architectures, Nat Commun., 11, 6240, 2020, 1-8.
 23. K. Manu, J. Jezierski, M.R.S. Ganesh, K.V. Shankar, S.A. Narayanan, Titanium in Cast Cu-Sn Alloys-A Review, Materials, 14, 16, 2021, 1-36.
 24. S. Kaiser, M.S. Kaiser, Impact of cold plastic deformation and thermal post-treatment on the physical properties of copper based alloys Al-bronze and α -brass. Acta Metall. Slovaca, 12, 2, 2021, 103-108.