

## **MICROSTRUCTURES AND MECHANICAL PROPERTIES OF 1100 Al CRYOROLLED ALLOY**

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### **ABSTRACT**

Influence of pre-treatment solution treatment of cryorolled 1100 Al alloy was investigated in the present work by employing microhardness test, tensile test and microstructural investigation using Vicker microhardness, Instron and optical microscope (OM). Samples were solution treated at various temperature (500°C for 6 hour, 540°C for 2 hour and 580°C for 2 hour) to study the effect of solution treatment on the cryorolled 1100 Al alloy sample and followed by post-annealing treatment. Sample solution treated at 540°C for 2 hours soaking time showed the highest increment of hardness value and strength by 34.67 % and 10.9 % respectively. The entire cryorolled samples showed severely deformed grain which are elongated along the rolling direction.

*Keywords: Aluminium 1100 alloy; solution treatment; cryorolling; post-annealing;*

### **INTRODUCTION**

Nanomaterials with ultrafine-grain (UFG) structures with grain size between 100 nm and 1  $\mu$ m have caused appreciable interest of material researchers for more than 50 years [1]. This interest is due to the superior mechanical properties such as higher strength, toughness, strength-to-weight-ratio, easy formability, high corrosion resistance and low ductility [2]. With the ever-growing demand for superior mechanical properties of aluminium alloy, severe plastic deformation technique (SPD) have been introduced to produce this alloy with ultrafine-grained microstructures [3].

The SPD methods make it possible to efficiently refine the grain size down to submicrocrystalline and nanocrystalline size range [4]. Different methods of SPD techniques such as equal-channel angular press [5], high pressure torsion, asymmetric rolling, grove pressing, and accumulative roll bonding have been used to develop ultrafine-grained bulk materials [6-7]. However, it requires high plastic strain for the grain refinement and also difficulties in producing long length products in UFG materials [8,9]. Conventional rolling route able to produce sheets or plates in large quantities for structural application. However, the conventional processing especially rolling route is a viable option [10]. However, due to high stacking fault energy in Al

alloy [11], conventional rolling process is not a suitable choice. To obviate this difficulty, rolling at cryogenic temperatures (cryorolling) has been identified to produce UFG materials from its bulk metals and alloys [12].

Cryorolling can be defined as rolling that is being done in a cryogenic (liquid nitrogen) condition which is below  $-190^{\circ}\text{C}$  [13]. The suppression of dynamic recovery during deformation at cryogenic temperature would lead to a larger accumulated dislocation density which can act as the potential recrystallization sites to produce UFG structure [14]. Taye et al. [15] have claimed that rolling under cryogenic temperature has been widely used to improve the materials properties especially the strength and hardness. Cryorolling has been studied mostly for stainless steel and aluminium alloy which has high tendencies for UFG production.

The development of UFG microstructure in aluminium is obtained by implementing a combined treatment of cryorolling and initial heat treatment such as annealing and solution treatment. Hence, it resulted in the combination of excellent strength, plasticity and ductility of the material [16]. The purpose of solution treatment is to take into solid solution the maximum practical amounts of the soluble hardening elements in the alloy. There should have sufficient length of time for solution treated in order to obtain a homogeneous supersaturated structure than followed by quenching [17].

According to Panigrahi et al. (2011), solute elements in the solid solution suppress the dynamic recovery during cryorolling of Al alloys. It is known that the presence of solute elements in solid solution of Al alloy during processing pins the dislocations and thus decrease the rate of dynamic recovery and refines the microstructure at high deformation strain. In order to refine the microstructure effectively, the second phase particles should be completely dissolved in matrix [3].

For addition, aluminum alloys that strengthened by cryorolling exhibited a reduction in ductility. Thus, it makes the material unsuitable for forming applications. Therefore, a suitable post treatment was given to the cryorolled materials to improve the ductility without sacrificing too much on the strength. Partial annealing or recovery annealing with temperature in the range of  $150\text{-}300^{\circ}\text{C}$  (below the recrystallization temperature) for at least 30 minutes can achieve the desired combination of strength and ductility [18].

Recent works of UFG metals mainly concentrated on the pre-treatment of 6000 and 5000 Al series. On the other hand, the pre-treatment of 1000 Al series has received only limited attention. Characterization of mechanical properties of combined pre-treatment and post treatment cryorolling process is also important for the engineering application. Therefore, the study of pre-treatment of 1100 Al alloy are expected to throw some light on the production of UFG material. The objective of the present work are, (i) to study the effect of solution treatment at different temperature before cryorolling process, (ii) to develop UFG 1100 Al alloy by cryorolling process combine with solution treatment and

post-annealing.

## EXPERIMENTAL

The 1100 Al alloy sheets (1 mm thick) with the composition of 0.57% Fe, 0.12% Cu, 0.13% Si, 0.03% Ti, 0.02% Mg, 0.013% Mn, 0.017% Ni, 0.01% Zn and 99.09% Al were cryorolled up to 30% thickness reduction. Three different temperatures of solution treatment; 500°C, 6 hours soaking time, 540°C, 2 hours soaking time and 580°C, 2 hours soaking time were selected based on previous finding [19] to study the effects of solution treatment before cryorolling. Cryorolling was carried out by dipping the solution treated sheet in liquid nitrogen for 30 minutes then followed by rolling in a double rolling mills for ten passes until 30% thickness reduction. Between successive passes, the processing sheet was immersed for 5 minutes in liquid nitrogen. The flow diagram of cryorolling process is shown in Fig 1. Then, the cryorolled samples were annealed at 250°C for 2 hours. Microstructures of selected samples were obtained by etching with Keller's reagent and were examined under Metallurgical Microscope Olympus BX51M. Microhardness of the samples was measured using Vicker Microhardness with 100 gf load measurement. At least five readings were taken on the flat surface of the sample parallel to the rolling direction. Tensile test were conducted using Instron Universal Testing Machine according to ASTM E8 standard with crosshead speed was constant at strain rate of 1 mm/min.

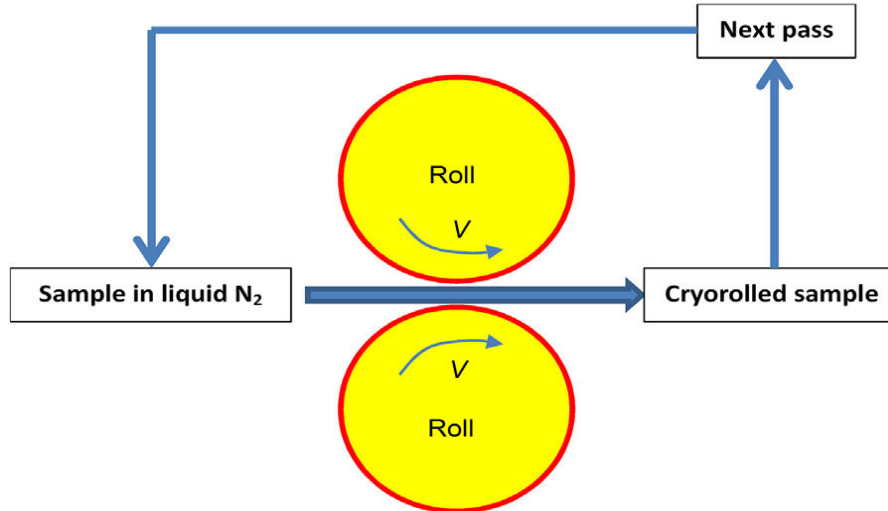


Figure1: The schematic illustration of cryorolling process (Yu et al., 2015)

## RESULT AND DISCUSSION

Based on the previous study, the proper heat treatment before cryorolling was identified to ensure the dissolution of solute elements into the solid solution prior to cryorolling. The heat treatment selected were 500°C with 6 hours soaking time, 540°C and 580°C with 2 hours soaking time. Fig 2 shows the microstructures of starting material of 1100

Al alloy and microstructures of solution treated sample at various temperatures.

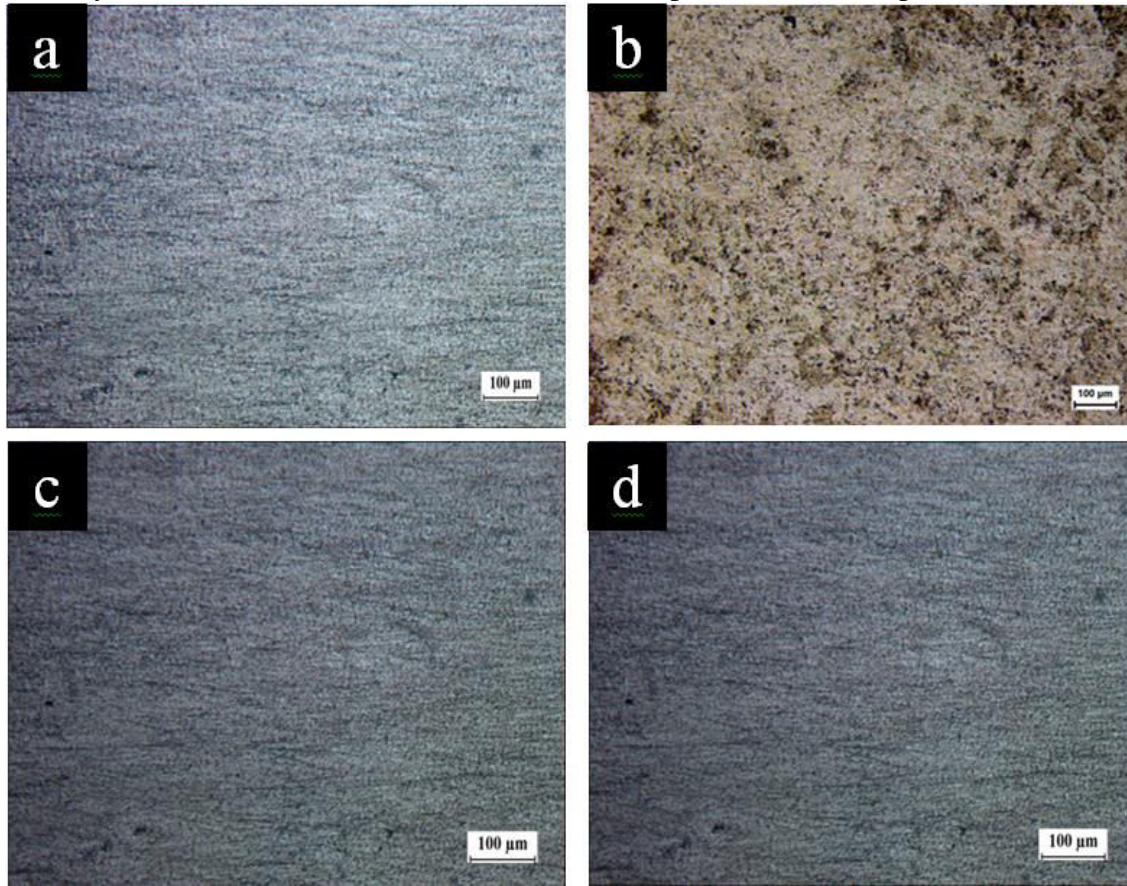


Figure 2: Microstructures of 1100 Al alloy after solution treatment at (a) raw material of 1100 aluminium alloy (b) 500°C for 6 h, (c) 540°C for 2 h and (d) 580 °C for 2 h

The samples solution treated at 500°C, 540°C and 580°C showed a homogenous structure of alloy. Sample solution treated at 500°C followed by cryorolling showed irregular elongated shape with average grain size of 45-50 µm. Meanwhile, both samples solution treated at 540°C and 580°C shows some elongated grains with fine grain size of 40-45 µm. Post treatment is important after deformation in order to improve ductility by reducing the dislocation density through recovery and recrystallization [20]. However, the improvement in ductility resulted in the reduction in strength. The effect of post annealing on the microstructures of the cryorolled sample is shown in Fig 3. Microstructure of cryorolled sample solution treated at 500°C after annealing showed formation of larger and coarser grains. Meanwhile, cryorolled sample solution treated at 540°C and 580°C followed by annealing showed small elongated grains. It can be observed that the microstructures of both cryorolled and post annealed samples are likely. The annealing helps in eliminating the residual stresses introduced during cyorolling and will relax the built up tension in the cryorolled material. According to Rangaraju et al. (2005) the post annealing resulted in sub-grain formation due to rearrangement of dislocations.

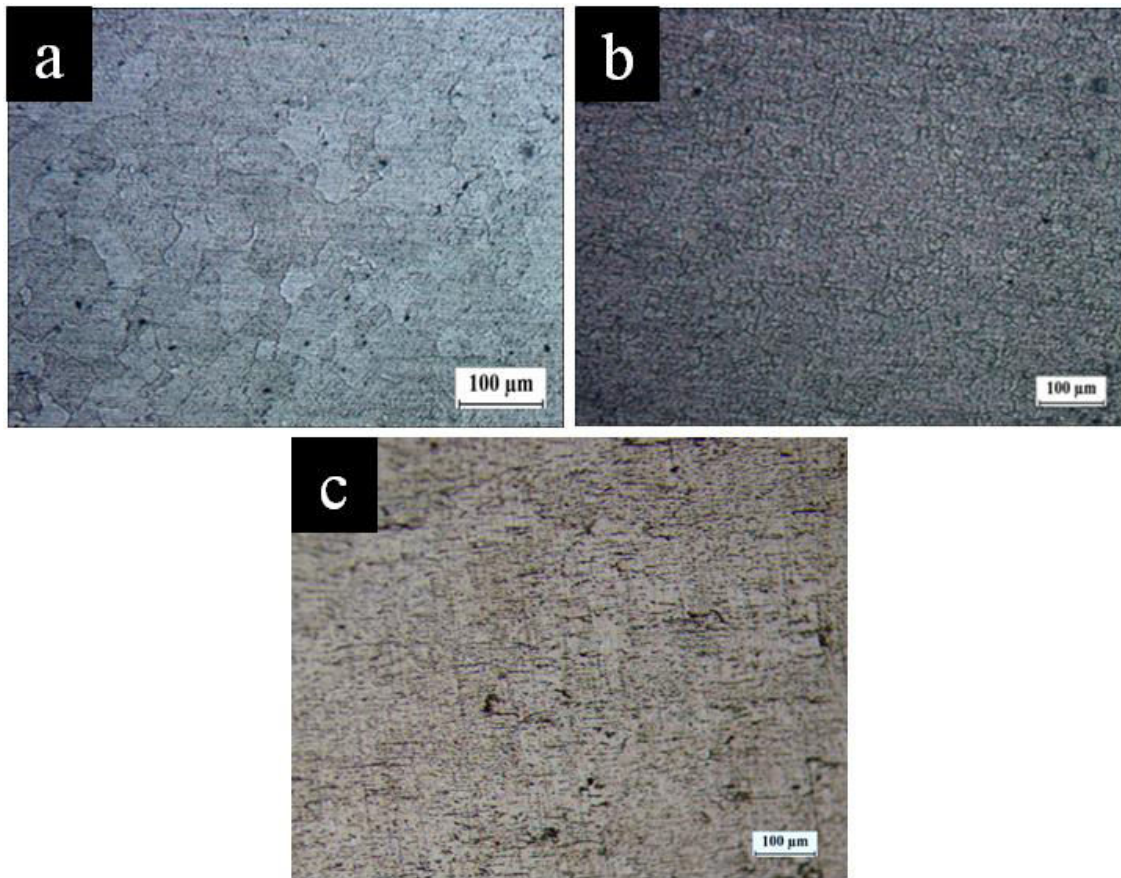


Figure 3: Microstructures of cryorolled sample solution treated at (a) 500°C for 6h, (b) 540°C for 2h and (c) 580°C for 2h followed by post annealing at 250°C for 2 hrs

Figure 3 shows the microhardness of 1100 Al alloy at different processing condition solution treated at various temperature. Cryorolled sample of solution treated sample at 540°C with 2 hours soaking time showed the highest increment in hardness value for 34.67 % compared to solution treatment sample. Meanwhile, the cryorolled sample of 500°C with 6 hours soaking showed 7.94 % and 580°C with 2 hours soaking time showed 26.12% increment in hardness value compare to solution treatment sample. In other hand, cryorolled+post annealing sample showed the reduction in hardness value compared with cryorolled samples (Fig 4). This is due to the post annealing that is subjected to the alloy after cryorolling. The decrease of the hardness value of cryorolled+post annealing sample for 500°C, 540°C and 580°C are 0.83%, 5.6% and 9.15% respectively.

Futhermore, all the cryorolled solution treated sample showed increment in strength (Figure 4) compare to solution treatment sample. Cryorolled sample at 540°C with 2 hours soaking time showed the the highest increment in strength of 10.9%. On other hand, cryorolled sample at 500°C and 580°C showed 2.93% and 2.73% increment of strength compared to solution treatment sample. The yield strength (Fig 5 and 6) of

cryorolled+post annealing sample is higher compared to cryorolled sample. The highest yield strength of cryorolled+post annealing sample achieved at solution treated at 540°C (88 MPa), while the lowest yield strength of the cryorolled+post annealing sample was at 500°C. Along with the improvement of hardness and strength, a significant improvement of ductility of 4 mm, 3 mm and 4 mm respectively achieved for cryorolled sample solution treated at 500°C, 540°C and 580°C.

The increment of strength and hardness value after deformation (cryorolling) is due to the cold temperature when dipped in liquid nitrogen. The cold temperature resulted in the contraction of aluminium matrix which reduced or closed the internal defects of the structure such as microporosities and vacancies which act to reduce the lattice parameter and leading to expulsion of the solute atoms and more volume of the second phase [15]. Furthermore, the combination of cryogenic temperature with rolling process has tremendous effect on the hardness of 1100 Al alloy. During dipping in liquid nitrogen, the volume of the material contracted due to cryogenic temperature. The volume contraction released great compression deformation energy that act as driving force for the formation and movement of the dislocation. The improvement in dislocation density played a significant role in enhancing the mechanical properties of the 1100 Al alloy [15]. Besides, dipping in liquid nitrogen helps to shrink the void and resulted in increasing the hardness of the material [16,17].

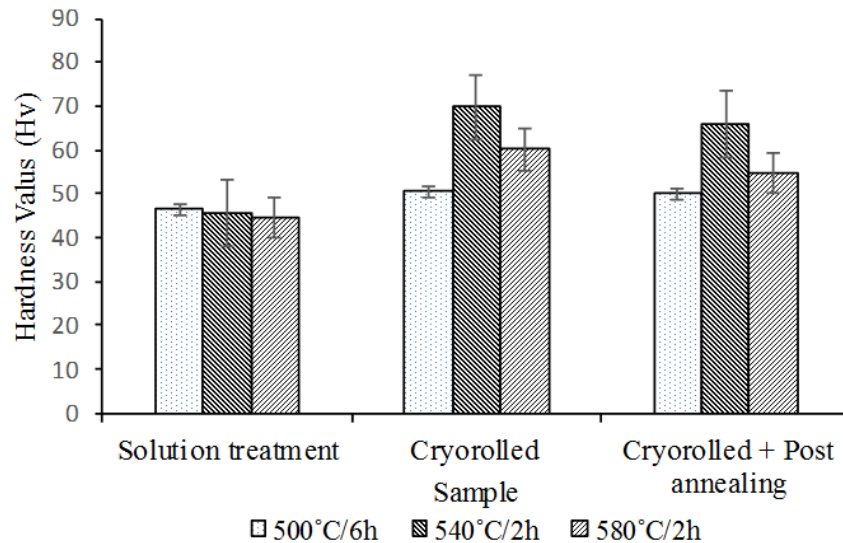


Figure 4: Vicker microhardness of 1100 Al alloy at different processing condition solution treated at various temperature

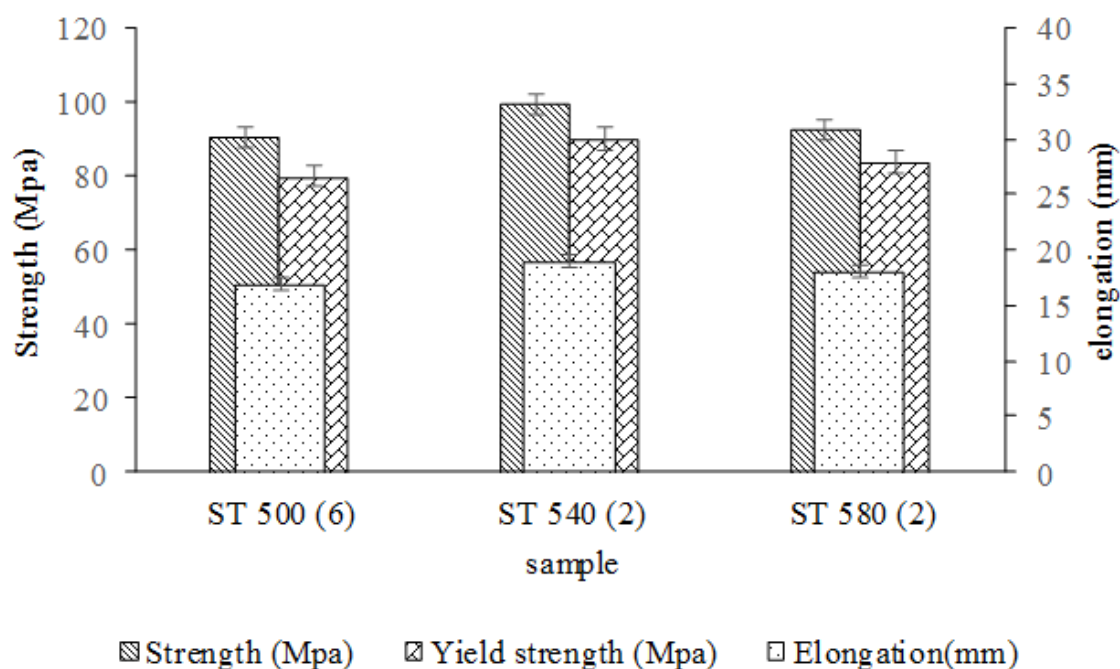


Figure 5: Tensile properties of cryorolled 1100 Al alloy at different processing condition solution treated at various temperatures

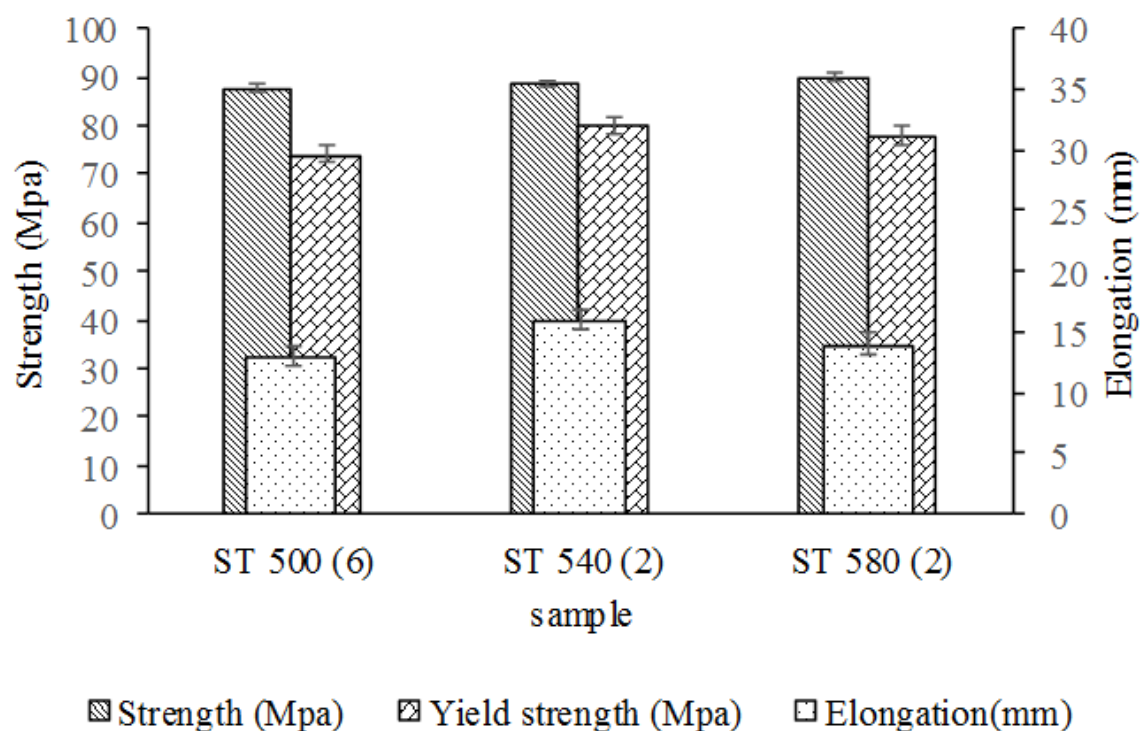


Figure 6: Tensile properties of 1100 Al alloy cryorolled at various solution treatment followed by post annealing

## CONCLUSION

From the present study of microstructure and mechanical properties of 1100 Al cryorolled alloy at various temperature of solution treatment and soaking time, 540°C with 2 hours soaking time showed the highest increment in hardness and strength. Solution treatment with proper temperature and soaking time play the important role for the production of cryorolled sample to ensure the dissolution of solute atom in the aluminium matrix and suppression of the dynamic recovery during cryorolling.

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