STUDY OF RECRYSTALLIZATION BEHAVIOR AND EFFECT OF HEAT TREATMENT ON ROLLED AND CRYOROLLED PURE COPPER

Submitted in partial fulfilment of the requirements of the degree of

Bachelor of Technology

in

Metallurgical and Materials Engineering

By

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Approval Sheet

This Project Work entitled “STUDY OF RECRYSTALLIZATION BEHAVIOUR AND EFFECT OF HEAT TREATMENT IN ROLLED AND CRYO ROLLED PURE COPPER ” carried out by Kilambi Vaishnavi Sahira (UG115124) and V.Sruthi (UG115151) is approved for the degree of Bachelor of Technology, , Metallurgical and Materials Engineering.

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Declaration

I declare that this written submission represents my ideas in my own words and where others' ideas or words have been included, I have adequately cited and referenced the original sources. I also declare that I have adhered to all principles of academic honesty and integrity and have not misrepresented or fabricated or falsified any idea/ data/ fact/ source in my submission. I understand that any violation of the above will be cause for disciplinary action by the Institute and can also evoke penal action from the sources which have thus not been properly cited or from whom proper permission has not been taken when needed.

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Certificate

This is to certify that the project work entitled "STUDY OF RECRYSTALLIZATION BEHAVIOR AND EFFECT OF HEAT TREATMENT ON ROLLED AND CRYO ROLLED PURE COPPER" is a bonafide record of work carried out by Kilambi Vaishnavi Sahira (UG 115124) , V. Sruthi (UG115151) submitted to the faculty of "Metallurgical and Materials Engineering Department", in partial fulfilment of the requirements for the award of the Degree of Bachelor of Technology in "Metallurgical and Materials Engineering" at National Institute of Technology, Warangal during the academic year 2014-15.

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ABSTRACT

Copper is an important engineering metal, since it is widely used in its unalloyed condition as well as in alloys with other metals. In the unalloyed form, it has an extraordinary combination of properties which make it basic material in the electrical industry, some of those properties being its high electrical conductivity and corrosion resistance, ease of fabrication, reasonable tensile strength, controllable annealing properties, and general soldering and joining characteristics. Copper can be deformed extensively upto 90%. The hardness of cryo rolled copper is more than rolled Copper. This indicates the advantage and superiority of cryo rolling over rolling.

In the present study Pure Copper is rolled and cryo rolled upto 90% deformation and will be heat treated at different annealing temperatures ranging from 150^{o}C to 300^{o}C and times ranging from 30 minutes to 3 hours. Characterization using Optical Microscope, SEM and Micro hardness tester will be done. Variation of micro structure and hardness with changing annealing temperature and annealing time will be analysed. The difference in the behavior of rolled and cryo rolled Copper will also be analysed. This study not only centres around the effect of heat treatment on the cold worked pure Copper but also explains partially recrystallization behavior using SEM studies. However a more detailed study can be done by characterizing the same using EBSD and DSC. In the present work results and necessary conclusions from micro structure photographs and hardness measurements will be made.

Key words : *Recrystallization, grain growth, micro hardness, cryo rolling*

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1 INTRODUCTION

1.1PURE COPPER:

Copper is an important engineering metal, since it is widely used in its unalloyed condition as well as in alloys with other metals. In the unalloyed form, it has an extraordinary combination of properties which make it basic material in the electrical industry, some of those properties being its high electrical conductivity and corrosion resistance, ease of fabrication, reasonable tensile strength controllable annealing properties, and general soldering and joining characteristics.

1.2 Applications of pure copper:

1. Copper is used in dental material.
2. Silver filling materials have 12% copper.
3. It is mostly used in structural part of automobile, e.g. carburetor.
4. Most of the electrical components are made of copper e.g. Wires.
5. Copper is very uncorrosive so doesn't rust easily. However, when it does rust, copper turns a blue-green colour. The statue of liberty is made out of copper.
6. Copper is also used in water pipes due to the fact that it doesn't corrode easily. This application is found in construction of monuments.
7. Copper is also used in currency.
8. It is also used in computer chips, integrated circuit boards and printed circuit boards.

1.3 Properties of pure copper:

1. Copper is a reddish coloured metallic metal.
2. It is the only other metal apart from gold that has a distinctive natural colour.
3. It has a high electrical and thermal conductivity. The only pure metal to have higher electrical conductivity is silver.
4. Its atomic number is 29.
5. Copper has a charge of 2+ and belongs to the transition metals.

6. It is used in electromagnets, motors, transformers and dynamos.
7. It is ductile and tough.
8. It is antibacterial.
9. It is easily mixed with other metals to form alloys such as bronze (copper and tin) and brass (copper and zinc).
10. It is one of the few metals to occur naturally as an uncompounded mineral.
11. It is non magnetic.
12. It can be easily joined. (Copper presentation by Ashleigh Barehm)

1.4 Recrystallization of pure copper :

It is known that the metals that have been hardened due to deformation will soften when annealed at a suitable temperature. The main process responsible for softening is recrystallization, which involves the generation of a new grain structure in the metal. Recrystallization is a process of fundamental importance in the thermo mechanical processing of metals since it restores a worked metal to an unworked and formable state. The most direct method to study the recrystallization behavior consists in determining the recrystallized fraction by optical microscopy. However, in heavily deformed copper, it is difficult to distinguish the deformed microstructure and the recrystallized microstructure. Consequently, other indirect methods have also been used to determine the recrystallized fraction. For example, hardness indentations, calorimetric measurements, neutron diffraction, electrical resistivity and electron back scattered diffraction (EBSD) techniques have also been employed for this purpose. The microstructures that evolve during recrystallization of deformed polycrystals are dependent upon the character of the deformed matrix (including stored energy content and local chemistry), the orientation relationship between the deformed structure and the growing grains, and the annealing temperature and ambient conditions. Of primary importance in this process are the local and neighboring lattice orientations and the dislocation density distribution. In low stacking fault energy materials, annealing twins develop that complicate the recrystallization process. The twinned structure generally alters the energy and mobility of a mobile interface, thereby either enhancing or retarding the growth of a given orientation. Many researchers have investigated the recrystallization process and have described oriented nucleation and oriented growth and their impact on the evolving microstructures. The effects of annealing twinning on recrystallization texture and the resulting grain boundary character distribution are to be known. Since texture and grain boundary structure affect various properties of polycrystalline metals, it is important to better understand these structures. The recrystallization kinetics in deformed pure copper has been investigated by various workers using differential scanning calorimetry (DSC) under isothermal conditions and in contrast, the non-isothermal recrystallization in this material is rarely discussed in the literature.

The recrystallization development of copper and its alloys have a strong scientific and industrial interest driven by the need to understand and control this phenomenon in order to optimize the properties through careful control of the thermo mechanical processing schedules.

1.5 Cold Rolling:

Rolling is the plastic deformation that occurs in a material when it is passed in between two rolls. Cold rolling is used to produce strip and sheets, Superior surface finish, Good dimensional tolerances, Strain hardening. The assumptions of cold rolling are:

1. No elastic deformation of the rolls

2. Elastic deformation of the sheet is negligible

3. Coefficient of friction is constant at all points

4. Plane strain condition

5. Deformation is homogeneous

6. Peripheral velocity of the rolls is constant

1.6 Cryo rolling :

Cryo rolling is performed at lower temperatures using liquid Nitrogen which is at a temperature of -190^{o}C. Cryo-rolling is a potential technique to improve strength and hardness of the material. It is a very effective and reliable process to get desired mechanical properties.

(a) Advantages of Cryo-rolling:

1. By cryo-rolling we can achieve a ultra-fine grain structure which improves a strength and ductility compared to cold rolling process

2.handling of the material is easy in cryo-rolling compared to hot rolling process

3.If subsequently we are doing a annealing process after cryo-rolling then we can get a desirable ductility

4.cryo-rolling require a less plastic deformation compared to sever plastic deformation process. From Sever plastic deformation process, we can also achieve an ultra fine grain structure, but it requires a large plastic deformation.

(b) Disadvantages of Cryo-rolling:

1. By doing only cryo-rolling, we cannot get a proper ductility. Subsequent annealing process is require, but it is a preferable

- For cryo-rolling process mostly we will use a Al & Cu material as it is a very soft and ductile material and it is not achieve easily a brittle structure at cryogenics temperature

1.7 Objectives of the present work

- To study the recrystallization behavior in rolled and cryorolled pure copper
- To observe variation of hardness, grains and micro structure .
- Characterization techniques

2. Literature Review :

Many researchers have investigated the recrystallization process and have described oriented nucleation and oriented growth and their impact on the evolving microstructures. Lucci et al. investigated the effect of the grain size of pure copper deformed by cold rolling on the activation energy and the kinetic parameters of recrystallization. On the other hand, most of the previous calorimetric studies of copper were concerned with the measure of the stored energy released during recrystallization process and the determination of the apparent activation of recrystallization process based on the variation of the linear heating rate.

2.1. Role of annealing twins during recrystallization

The microstructures that evolve during recrystallization of deformed polycrystals are dependent upon the character of the deformed matrix (including stored energy content and local chemistry), the orientation relationship between the deformed structure and the growing grains, and the annealing temperature and ambient conditions. Of primary importance in this process are the local and neighboring lattice orientations and the dislocation density distribution. In low stacking fault energy materials, annealing twins develop that complicate the recrystallization process. The twinned structure generally alters the energy and mobility of a mobile interface, thereby either enhancing or retarding the growth of a given orientation. Many researchers have investigated the recrystallization process and have described oriented nucleation and oriented growth and their impact on the evolving microstructures. The effects of annealing twinning on recrystallization texture and the resulting grain boundary character distribution are of great importance. Since texture and grain boundary structure affect various properties of polycrystalline metals, it is important to better understand these structures.

2.2 Cold deformation:

Cold deformation plays an important role in that it provides the crystalline defects and the stored energy to control the final grain size during subsequent recrystallisation. During annealing, the defects act as a source for nucleation while the stored energy in the material is the driving force for recrystallisation.

Cold deformation can also be used to modify the precipitation behaviour in metastable alloys in order to achieve high densities of fine α precipitates during ageing.

Cold deformation has a severe effect on microstructure of metals. Firstly, the changes in grain morphology result in an increase in the total grain boundary area. Secondly, the appearance of an internal substructure comprising micro bands, shear bands and sub-grains within the grains is the result of the net accumulation of dislocations.

2.3 Annealing:

Annealing is a heat treatment process to reduce residual stresses. During the annealing of cold deformed materials, the process of recovery, recrystallisation and grain growth take place concurrently.

2.4 Recovery:

Recovery can happen either during deformation at high temperatures (dynamic recovery) or during annealing (static recovery). It is defined as the process by which deformed grains can reduce their stored energy via the removal and rearrangement of dislocations that accumulate during heating. A change in microstructure during recovery is relatively inhomogeneous and does not influence the high-angle grain boundaries between deformed grains. Changes in mechanical properties are characterized by a decrease in strength (softening) and an increase in ductility due to the reduction of dislocation densities through recovery.

2.5 Recrystallization:

Discontinuous recrystallizationis described as the process by which new grains nucleate and grow to consume the originally deformed grains. It is different from recovery in that the microstructural changes are associated with the formation and migration of high angle grain boundaries and that these processes are driven by stored energy of deformation and temperature of annealing. A significant decrease in strength and an increase in ductility occur during recrystallization, such that it is more prominent effect in comparison to recovery. On the other hand, continuous recrystallization occurs uniformly with a discernible nucleation stage. Recrystillization may also occur either during deformation at high temperatures (dynamically) or during annealing after deformation (statically).

2.6 Grain growth

Grain growth is the process in which an increase in grain size occurs via a curvature driven migration of high angle grain boundaries resulting in an overall decrease in boundary area. The difference between the recrystallization and grain growth phenomena is the driving force. Whereas, the driving force for the recrystallization process is the stored energy of dislocations introduced during deformation that for the grain growth is the high angle grain boundaries around their immediate vicinity. Abnormal grain growth is defined as that process of secondary recrystallization under which grain coarsening is inhomogeneous and a few isolated grains grow at the expense of the average recrystallised matrix.

2.7 Cryo rolling and rolling data of copper:

In this work, we use cold rolling, a common, inexpensive industrial process, to impart large plastic strains to Cu. Such a simple processing route is usually not ideal for producing nanostructures in metals. Cold rolling of Cu at RT has been studied extensively before, with thickness reduction typically in the range of 0-15,000% (Von Mises equivalent strain = ¼ 0-5). At medium to high plastic strains ($1 < \gamma < 5$), it is generally understood that multiple dislocations interact, forming multifarious defects such as dislocation cells, cell blocks, micro bands, twins and sub grains. Nano crystallites with high angle grain boundaries are usually not found. At extremely high strains ($\gamma > 5$), data are scarce because the rolled sheets start to break up and intermediate annealing is necessary before further cross sectional reduction is possible. Commercial Cu can be rolled to very large plastic strains ($\gamma > 5$) without cracking even at cryogenic temperatures. After cryo-rolling and microstructure evolution was found to be a complex process involving mainly geometrical effects associated with strain and discontinuous recrystallization but also including limited twinning and grain subdivision. Recrystallization was deduced to be static in nature and probably occurred during static storage of the material at room temperature after cryogenic rolling. (Dynamic Processes for Nanostructure Development in Cu after Severe Cryogenic Rolling Deformation by Yinmin Wang, Tong Jiao and En Ma., Materials Transactions, Year : 2003). Pure copper strips with initial cube texture were subjected to a double rolling process (deformation amount ranges from 50% to 95%), and the surface textures evolution law and mechanism of double-rolled strips were studied by an X-ray diffraction technique. The results indicated conditions for favourable texture of pure copper to obtain beneficial grain growth. (Mechanism of Surface Texture Evolution in Pure Copper Strips Subjected to Double Rolling Xiyong Wang, Xuefeng Liu, Jianxin Xie Progress in Natural Science: Materials International Volume 24, Issue 1, February 2014, Pages 75–82).

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3 EXPERIMENTAL WORKS:

3.1 Material:

The material taken for this project is Commercial pure Copper which is procured from market.

3.2 Cold Rolling and Cryo-Rolling:

The as received sample is first well polished and etched to observe microstructure under optical microscope. Then it is rolled to 50%, 70% and 90% deformation and also it is cryorolled for 50%, 70% and 90% deformations and microstructure is observed in all cases. It is a very common technique in manufacturing and is the desired process in the fabrication of brackets and bent parts. The strain (stretching) that is applied to samples is also a form of cold work. Cold work has a significant effect on the mechanical properties of alloys and predictions can be made concerning the properties if the amount of cold work is known. Cold working is the process of strengthening of a metal by plastic deformation. This strengthening occurs because of dislocation movements and dislocation generation within the crystal structure of the material.

The basic principle is that, when a piece of metal is rolled in between two rolls, the thickness is reduced as a result of the compressive stresses exerted by the rolls and it can be treated as a two-dimensional deformation in the thickness and length directions neglecting the width direction. This is due to the fact that the length of contact between the rolls and work piece is generally much smaller than the width of the sheet passing through and the undeformed material on both sides of the roll gap is restraining the lateral expansion along the width direction.

The metal piece experiences both vertical and horizontal stresses caused by the compressive load from the rolls and the restrains by the portions of the metal piece before and after the material in contact with the roll respectively.

As the rolls exert a vertical stress on the metal piece, the metal piece exerts the same amount of stress back onto the rolls itself. As such the rolls are subjected to elastic deformation due to this stress induced by the work piece. As a result the mechanical properties of the cold rolled alloy can be improved.

The grains of the resultant cold rolled alloy are comparatively elongated in the direction of rolling as we can observe in the microstructure.

3.2.1 Cold rolling:

It is a process by which the sheet metal or strip stock is introduced between rollers and then compressed and squeezed. The amount of strain introduced determines the hardness and other material properties of the finished product. The advantages of cold rolling are good dimensional accuracy and surface finish.

3.2.2 Cryo-Rolling :

Cryogenic rolling is one of the potential techniques to produce nano structured bulk materials from its bulk counterpart at cryogenic temperatures. It can be defined as rolling that is carried out at cryogenic temperatures. Nanostructure materials are produced chiefly by severe plastic deformation processes. The majority of these methods require large plastic deformations. In case of cryorolling, the deformation in the strain hardened metals is preserved as a result of the suppression of the dynamic recovery. Hence large strains can be maintained and after subsequent annealing, ultra-fine-grained structure can be produced. It is supposed that at very low temperatures of deformation initially grain growth is prevented. Then by hindering the distributions of dislocations, density and development of corresponding internal stresses takes place. This leads to further refinement of microstructure. (Effect of Cryogenic Temperature Rolling on Mechanical Properties and Microstructure of Pure Copper by Kandarp Changela, H.B Naik, K.P. Desai.).

In cryo-rolling the material is dipped in liquid nitrogen (-190^{o}C) and held it for 30 minute or one hour (depending on our requirement) and the material is rolled between two rollers. Rolling and cryo rolling were both done in the mechanical metallurgy lab of metallurgy and materials science department of NIT Warangal.

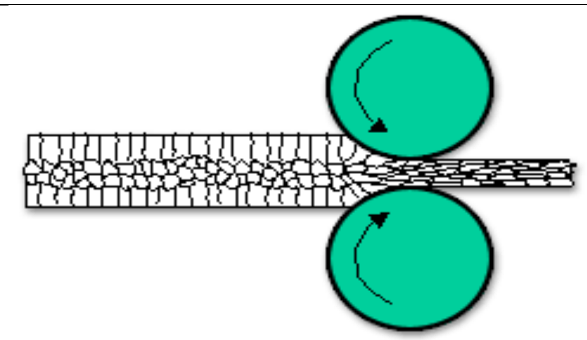


Figure 3.1: 2-High cold rolling mill.

ROLLED SAMPLES

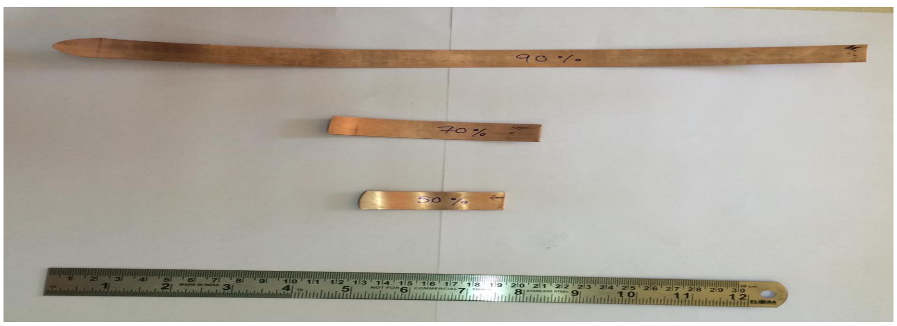


Figure 3.2 Rolled samples

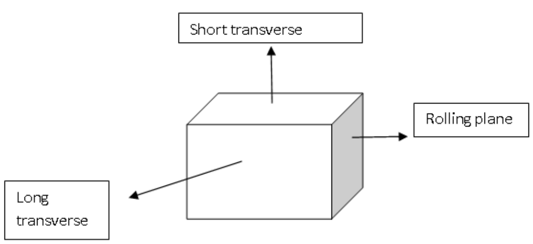


Figure 3.3 : Rolling plane demonstration

CRYO ROLLED SAMPLES :

FIGURE 3.4 CRYO ROLLED

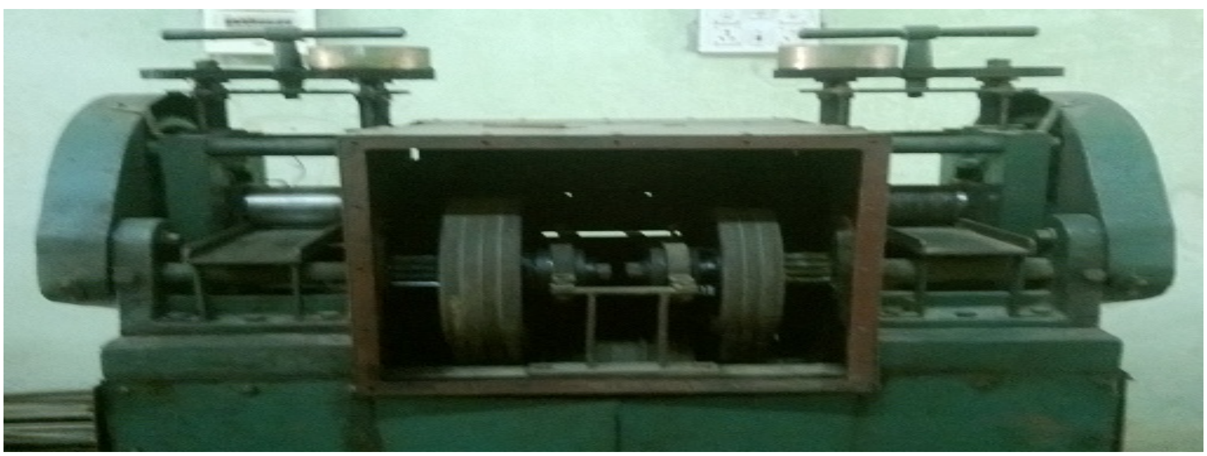


Figure 3.5 COLD ROLLING MILL IN MME, DEPARTMENT AT NITW

3.3 Heat treatment:

The samples were heat treated in furnace and air cooled to observe recrystallization behavior. They are heat treated at 150C, 200C, and 300 C for 30minutes, 1 hour and 3 hours each. Thus a matrix of microstructures is analyzed for each deformation.

Figure 3.6: Electric muffle furnace used for heat treatment

3.4 SAMPLE PREPARATION AND METALLOGRAPHY:

Metallography consists of the study of the constitution and structure of metals and alloys.

Proper preparation of metallographic specimens to determine microstructure and content requires a rigid step-by-step process to be followed. In sequence, the steps include sectioning, mounting, course grinding, fine grinding, polishing, etching and microscopic examination. Specimens must be kept clean and preparation procedure should be carefully followed in order to reveal accurate microstructures. The samples for the micro-structural examination were prepared by the conventional polishing technique on four different grits of polishing papers i.e. 1/0 , 2/0, 3/0, 4/0. Disc polishing on velvet cloth using diamond paste as lubricant was done. Finally the samples were etched. The sample preparation involves the following steps which are explained below :

Sectioning involves Separate test pieces or coupons attached to castings or forgings so that a minimum of sectioning is required for producing metallographic specimens.

Cutting is done to the samples for example in this case pressure tube samples to produce samples in rolling, transverse and normal directions. Each sample consisted of a short transverse, long transverse and rolling plane. The grain orientations vary accordingly. The samples are cut using abrasive wheel cutters etc preserving the axes and noting the sections formed after cutting.

Mounting is essential for small specimens as it supports the specimen in stable medium for grinding, polishing etc. The majority of metallographic specimen mounting is done by encapsulating the specimen into a compression mounting compound (thermosets - phenolics, epoxies, diallyl phthalates or thermoplastics - acrylics), casting into ambient castable mounting resins (acrylic resins, epoxy resins, and polyester resins), and gluing with a thermoplastic glues.

Paper polishing is done on Silicon Carbide papers (of grades 320, 500, 1000, 1200, 2400 etc) on an automatic polishing machine to achieve better results with less labor.

Automatic Polishing machine is used for high quality preparation of most metallographic specimens. Automatic polishing equipment usually allows the preparation of several specimens simultaneously.

Diamond polishing consists of polishing the sample using 1µm diamond paste and kerosene to obtain mirror finish. Kerosene is used as it acts as a coolant as well as dispersive medium for the paste.

Aluminium polishing is done using Aluminium Nitrate solution. It is a polishing abrasive and is used for getting a surface with mirror finish. Water must be dripped while polishing.

Colloidal silica polishing is carried out on the Tegradoser Polishing Machine (Tegrapol-21) using colloidal silica suspension for micro scratch reduction. Colloidal silica polishing suspensions are unique because they provide both a dispersing action as well as a chemical mechanical polishing (CMP) action. It is effective as an additive for the intermediate diamond polishing of metals and is also the best polishing abrasive for eliminating subsurface and surface damage in ceramics because of its CMP polishing action.

Colloidal silicas are suspensions of fine amorphous, nonporous, and typically spherical silica particles in a liquid phase.

The metallographic equipment that was used for grinding and polishing is shown in the following Figure.

Figure 3.7: Belt grinder.

Figure 3.8: Polishing machine.

<u>Chemical composition of etchant:</u>

95ml Distilled water

2.5ml nitric acid

1.5ml $FeCl_3$

3.5 Material Characterization:

3.5.1 Optical Microscopy:

Optical microscopy is performed to observe the microstructural features like grains, grain boundaries and approximate grain size. It is used as a quick method of observing the effect of processing parameters on the microstructure rather than using it for obtaining conclusive experimental results. The samples are observed under magnifications of 50x, 100x and 200x and 500X. Then, the samples were observed under optical microscope and the micrographs were captured at different magnifications ranging from 10x to 100x. The machine had four imaging modes:

1. Polarized Light (POL)

2. Differential Interface Contrast (DIC)

3. Bright Field (BF)

4. Dark Field (DF)

The optical microscope is shown in the following Figure.

Figure 3.9: Optical microscope.

3.5.2 Scanning Electron Microscopy

A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that can be detected and that contain information about the sample's surface topography and composition. The electron beam is generally scanned in a raster scan pattern, and the beam's position is combined with the detected signal to produce an image. SEM can achieve resolution better than 1 nanometer. Specimens can be observed in high vacuum, in low vacuum, and (in environmental SEM) in wet conditions.

The scanning electron microscope employs beam of electrons directed at the specimen. It consists of electron gun, lenses, and condenser and vacuum systems. It is primarily used to study the surface or near surface of bulk specimens. It is much easier for naked eye to interpret this type of image produced by SEM.

The electron source is usually of the tungsten filament.

The samples were observed under SEM (model: VEGA3 TESCON)with a working distance of 14.5mm and the micrographs were captured at different magnifications ranging from 500x to 8000x at a speed of 7(100.00µsec/pixel) and the results obtained with the 2500x and 3000x magnifications are presented in this report.

3.5.3 Micro Vickers hardness test :

Vickers hardness test is widely used to evaluate mechanical properties of materials because of its simple technique. The Vickers hardness test adopts diamond pyramid indenter which makes geometrically similar depressions regardless of size. The hardness therefore represents the index of a stress at a given strain. The effect of severe cold-working on the properties, particularly on the hardness, of metals such as copper has been the subject of extended discussion. It is common knowledge that moderate amounts of coldwork materially increase the hardness and tensile strength of many metals, but that an excessive amount of cold-work may result in the development of internal or external structural defects which seriously impair the mechanical properties.

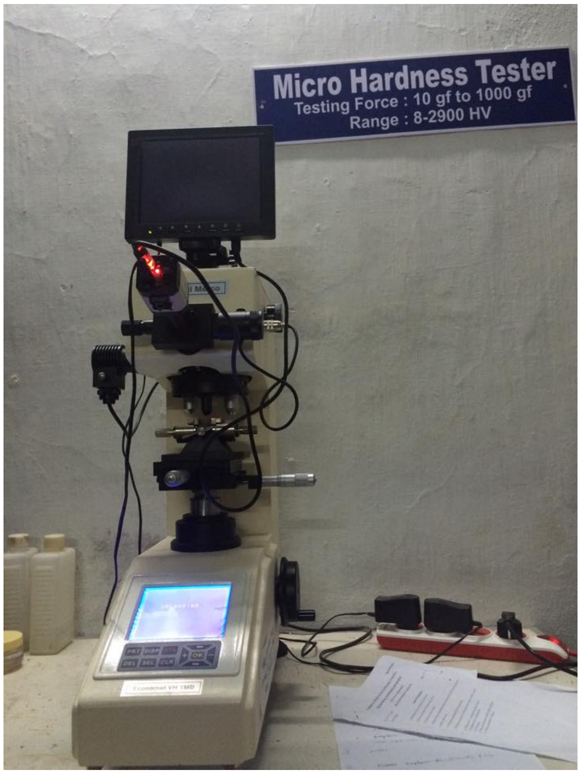


Figure 3.10 Micro hardness tester

Micro Vickers hardness was carried out in the micro hardness tester available at Mechanical Department.

Figure 3.11 Indentation of micro hardness tester on the sample

3.5.4 Differential Scanning Calorimetry (DSC)

Differential scanning calorimetry or DSC is a thermoanalytical technique in which the difference in the amount of heat required to increase the temperature of a sample and reference is measured as a function of temperature. Both the sample and reference are maintained at nearly the same temperature throughout the experiment. The basic principle underlying this technique is that when the sample undergoes a physical transformation such as phase transitions, more or less heat will need to flow to it than the reference to maintain both at the same temperature. Whether less or more heat must flow to the sample depends on whether the process is exothermic or endothermic.

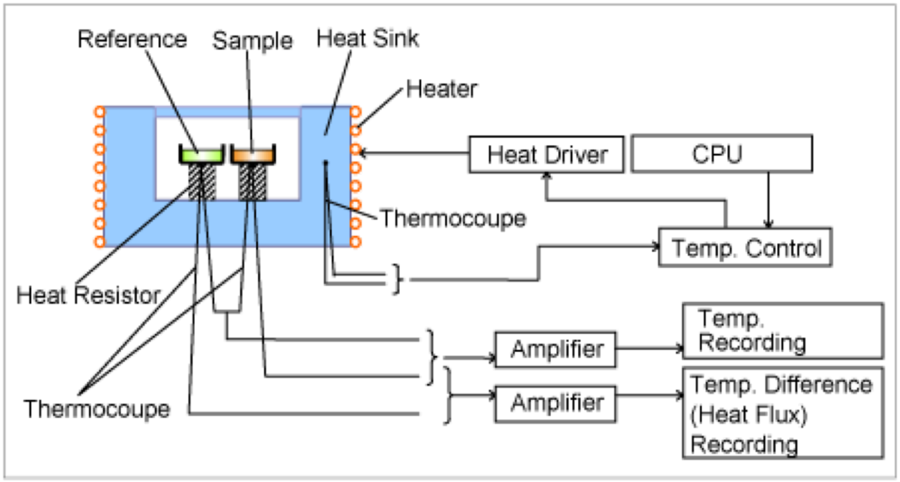


Figure 3.12 Block Diagram of Differential Scanning Calorimeter

Characterization of samples:

Micro Vickers hardness of all the samples of as-received, rolled, cryorolled and heat treated samples were taken.

SEM scans of 300C at 1 hour and 3 hours and 150 C at 3 hours are taken. Grain size using line intercept method is calculated from obtained micrographs. At constant temperature and varying time i.e.., 300C and at 1 hour and 3 hours SEM scans were taken to compare the grain size.

DSC was done to measure recrystallization kinetics. Micro hardness and DSC scans are used to estimate activation energy and predict recrystallization kinetics.

4 RESULTS AND DISCUSSIONS :

% of deformation on rolling	Initial thickness	Final thickness
50	6.69	3.35
70	6.7	2.01
90	6.65	0.67

% of deformation on cryo rolling	Initial thickness	Final thickness
50	6.69	3.36
70	6.65	2.05
90	6.76	0.60

Table 4.1 : thickness of rolled and cryo rolled samples

4.1 Optical images of heat treated and rolled and cryo rolled samples as observed on rolling plane :

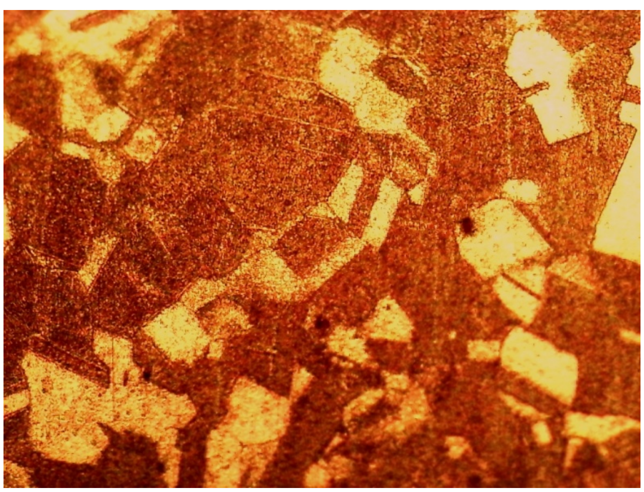


Figure 4.1 As received pure Copper at 200X

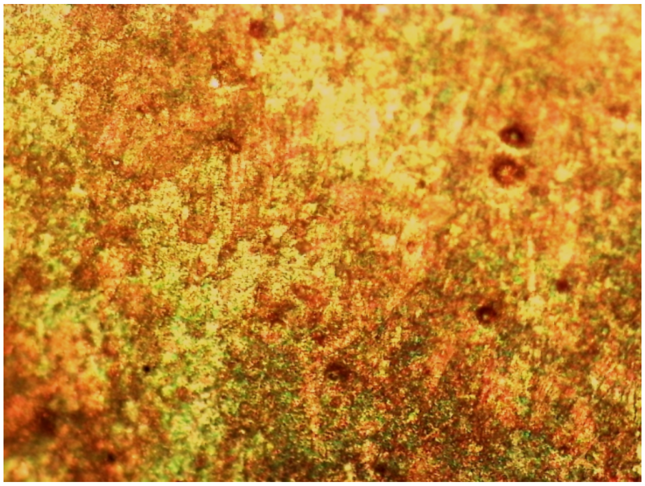


Figure 4.2 At 200x 50% rolled pure

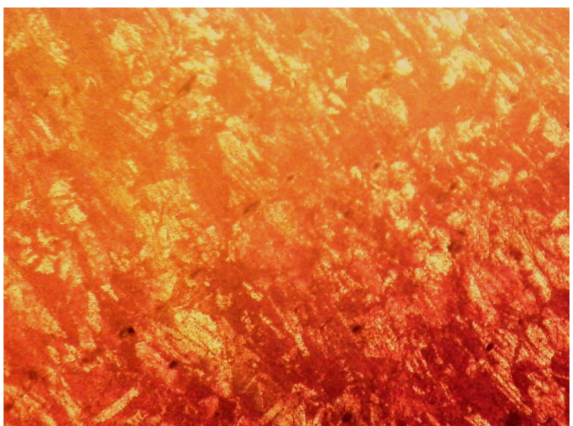


Figure 4.3 At 200X 50% cryo rolled

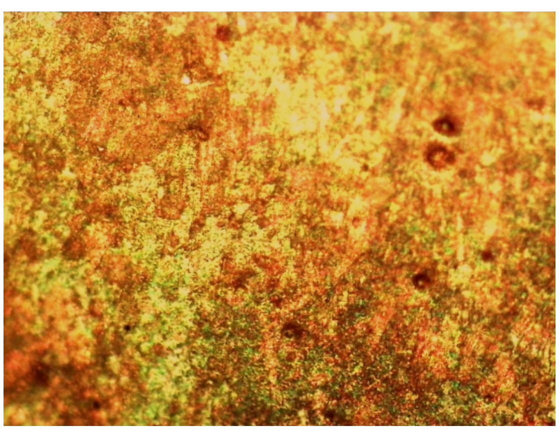


Figure 4.4 At 200X 70% rolled pure

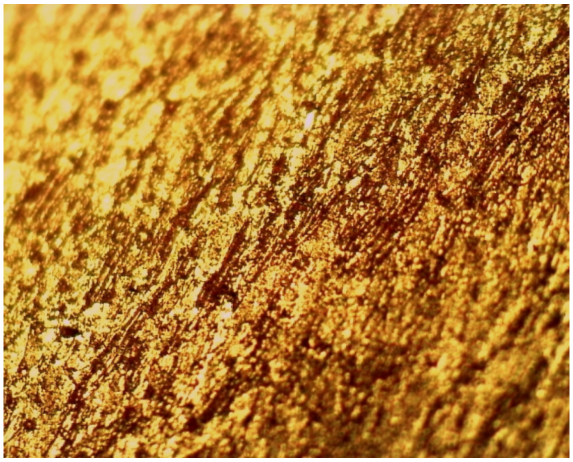


Figure 4.5 At 200X 70% cryo rolled pure

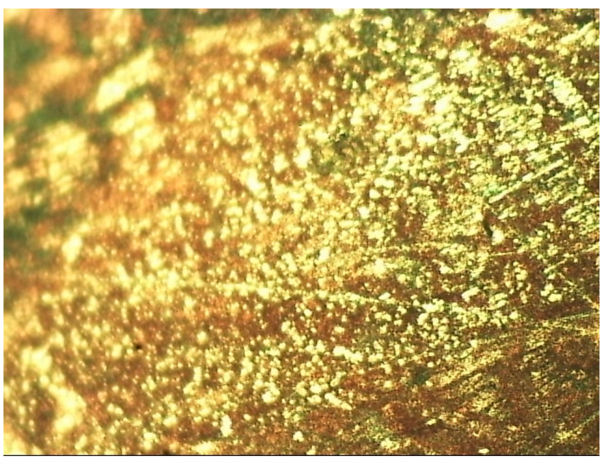


Figure 4.6 150°C, 30 minutes, 50%

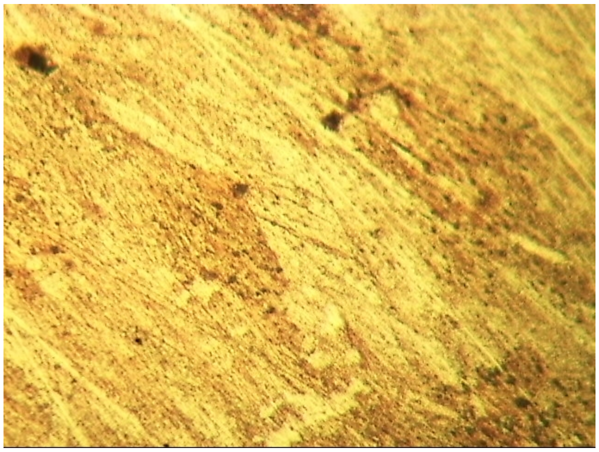
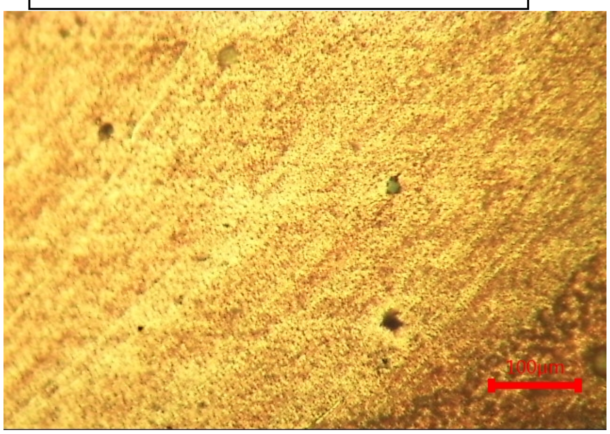


Figure 4.7 : 150°C, 1 hour, 50% Cryo



150°C, 1 hour, 90% cryo rolled

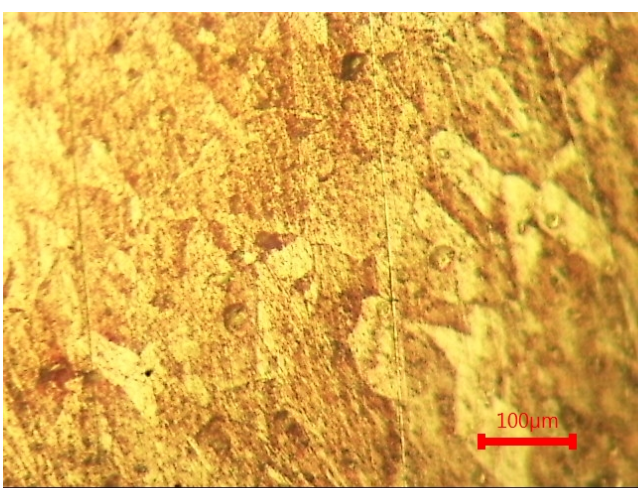


Figure 4.8 : 150°C, 1 hour, 50% rolled

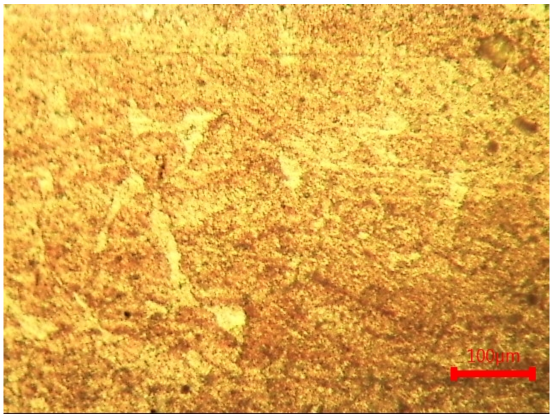


Figure 4.9 : 150ºC, 1 hour, 90% rolled

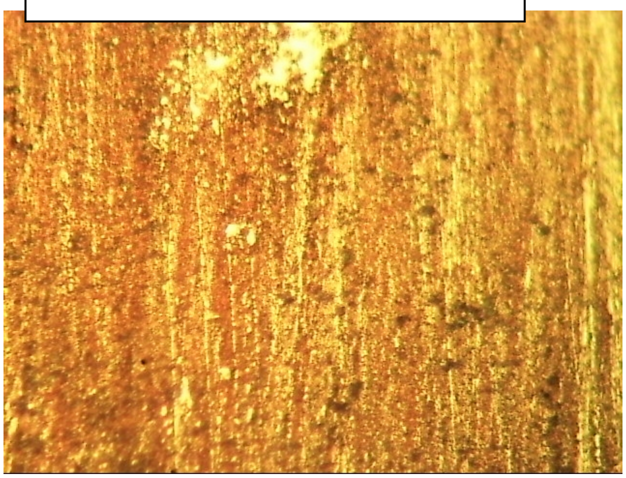


Figure 4.10 : 150ºC, 3 hours, 90% cryo

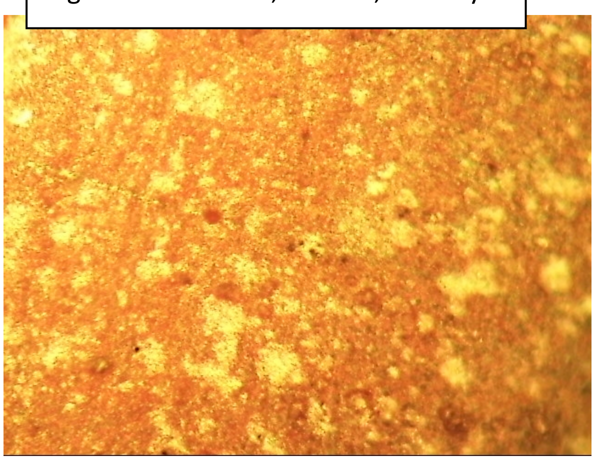


Figure 4.11 : 150ºC, 3 hours, 90% rolled

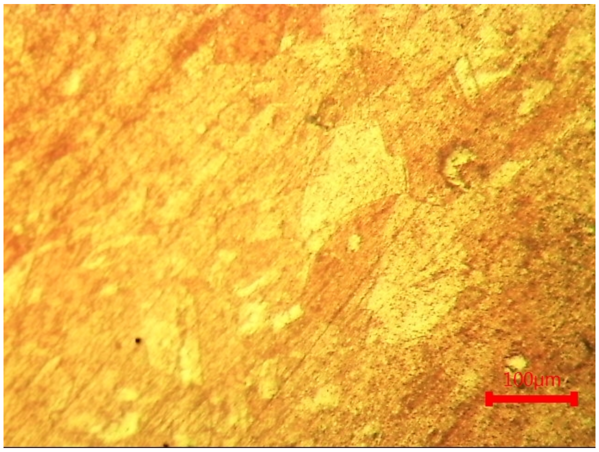


Figure 4.12 : 300ºC, 1 hour, 50% rolled

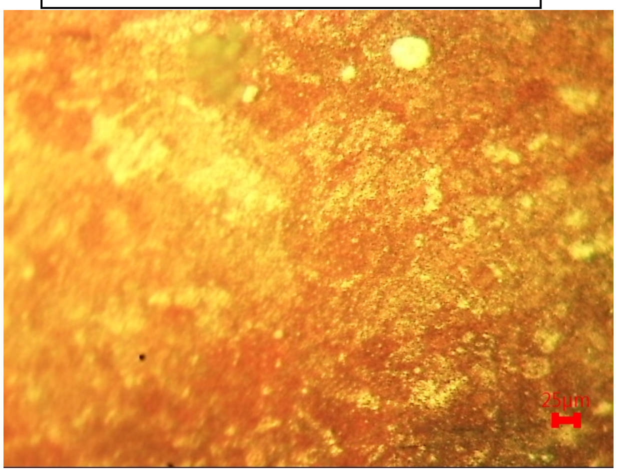


Figure 4.13 : 300ºC, 1 hour, 90% rolled

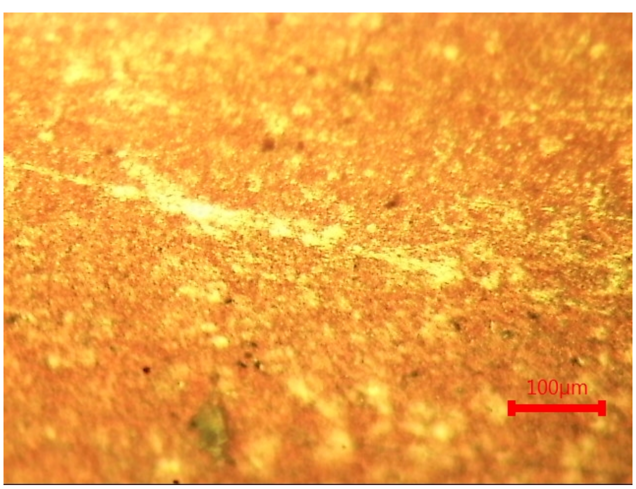


Figure 4.14 : 300ºC, 3 hours, 50% cryo

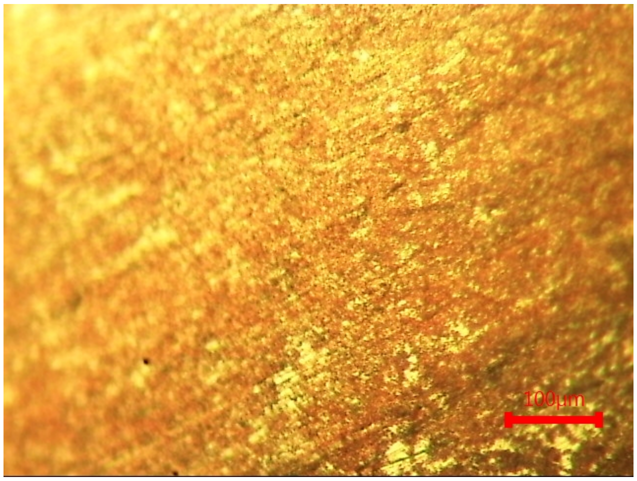


Figure 4.15 : 300ºC, 3 hours, 90% cryo

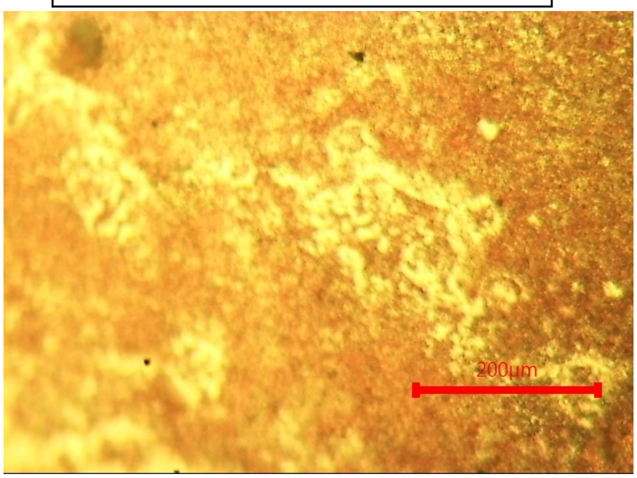


Figure 4.16 : 300ºC, 3 hours, 90% rolled

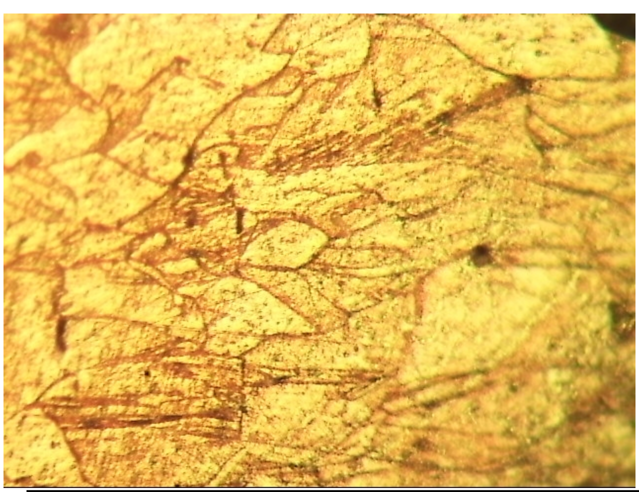


Figure 4.17 : 300ºC, 30 minutes, 50% cryo rolled

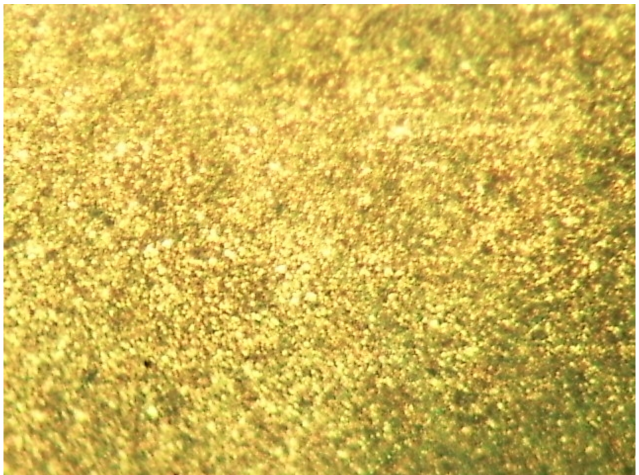


Figure 4.18 : 300°C, 30 minutes, 50% rolled

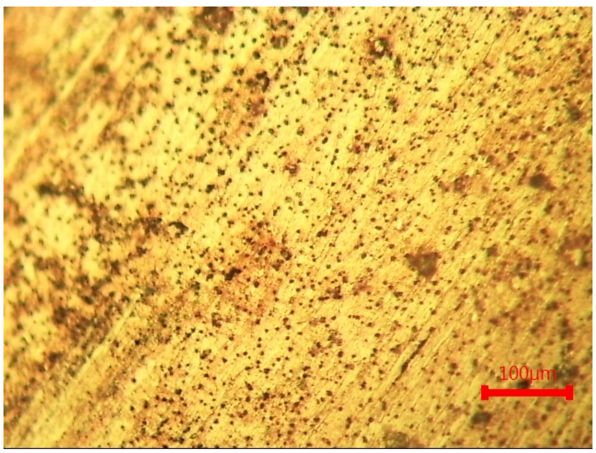


Figure 4.20 : 300°C, 30 minutes, 90% rolled

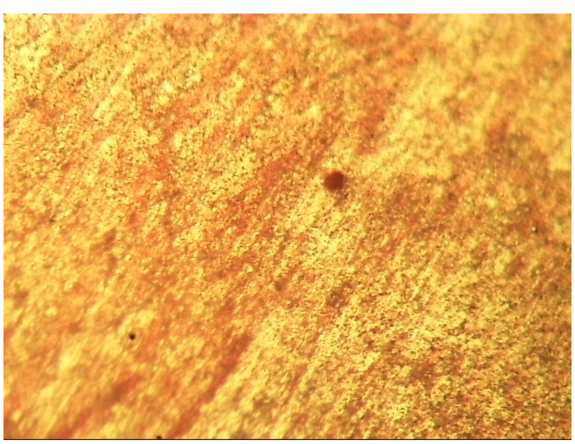


Figure 4.21 : 300°C, 30 minutes, 90% cryorolled

4.2 SEM images of heat treated samples as observed on rolling plane:

Figure : 4.22 : Sem images of 150^{o}C, 3 hours, Cryo rolled pure Copper samples which are 90% cryo rolled at different magnifications i.e.., 2kx, 2.5kx and 3 kx respectively:

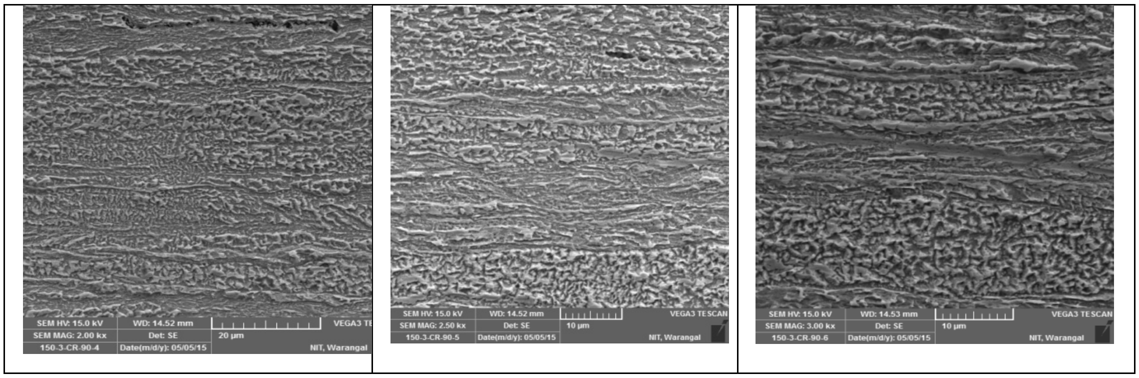


Figure 4.23 : Sem images of 150^{o}C, 3 hours, Rolled Copper pure Copper samples which are 90% deformed at different magnifications i.e.., 1.5kx, 2kx, 2.5kx respectively :

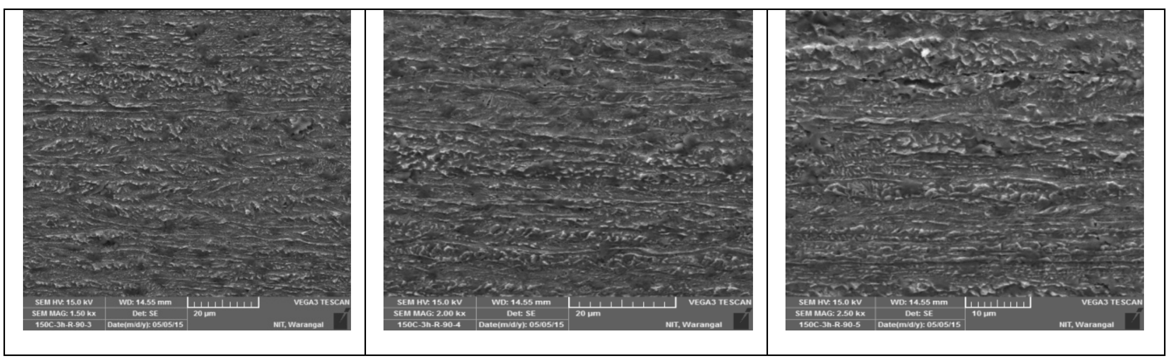


Figure 4.24 : Sem images of 300^{o}C, 30 minutes cryo rolled pure Copper upto 90% deformation at different magnifications i.e.., 2kx, 2.5kx and 3 kx:

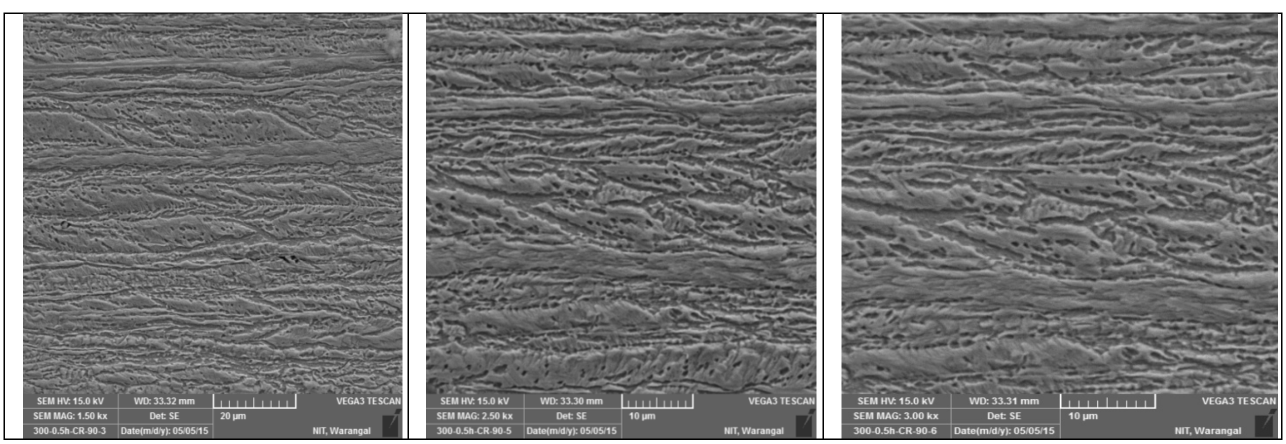


Figure 4.25 : Sem images of 300ºC, 30 minutes rolled pure Copper upto 90% deformation at different magnifications i.e.., 1.5kx, 2kx, 2.5kx respectively

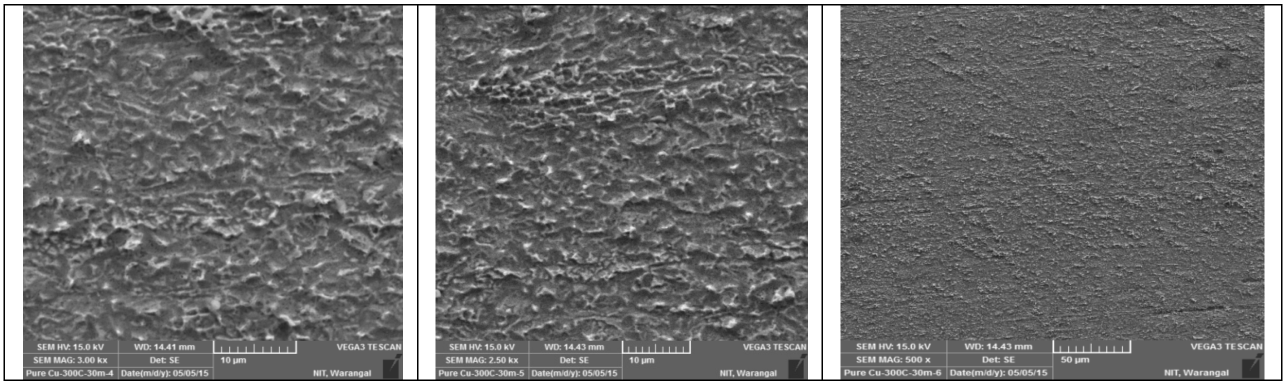


Figure 4.26 : Sem images of 300ºC, 3 hours, cryo rolled pure Copper upto 90% deformation at different magnifications i.e…, 1.5kx, 3kx, 5kx respectively.

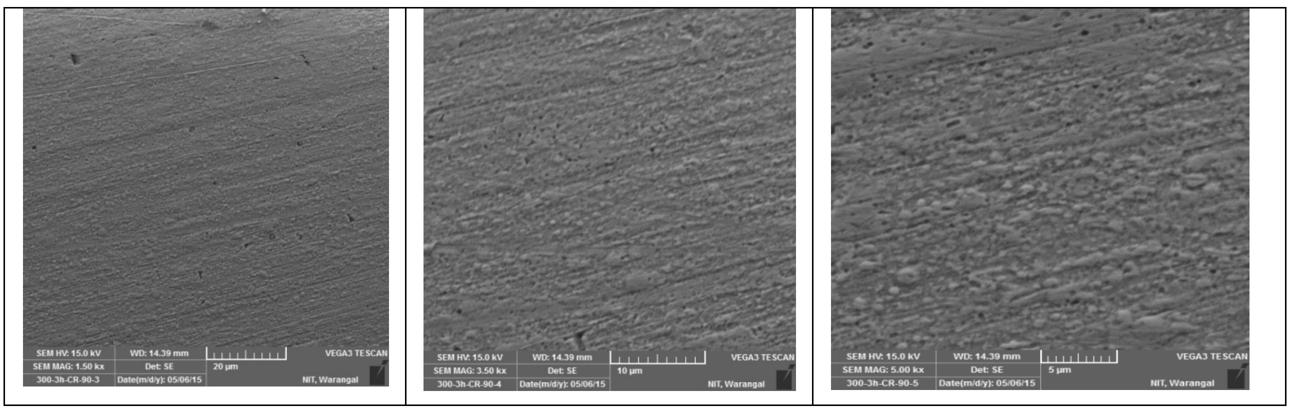
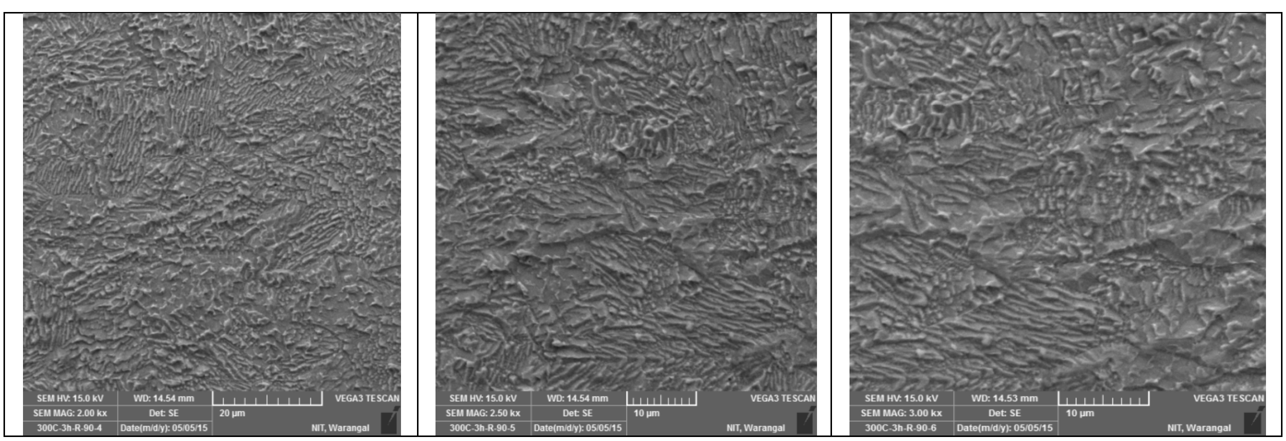


Figure 4.27 : Sem images of 300ºC, 3 hours rolled pure Copper at 90% deformation at different magnifications i.e…, 2kx, 2.5kx and 3 kx respectively.



4.3 Hardness measurements :

ROCKWELL HARDNESS VALUES(Rb):

Load=100kgf

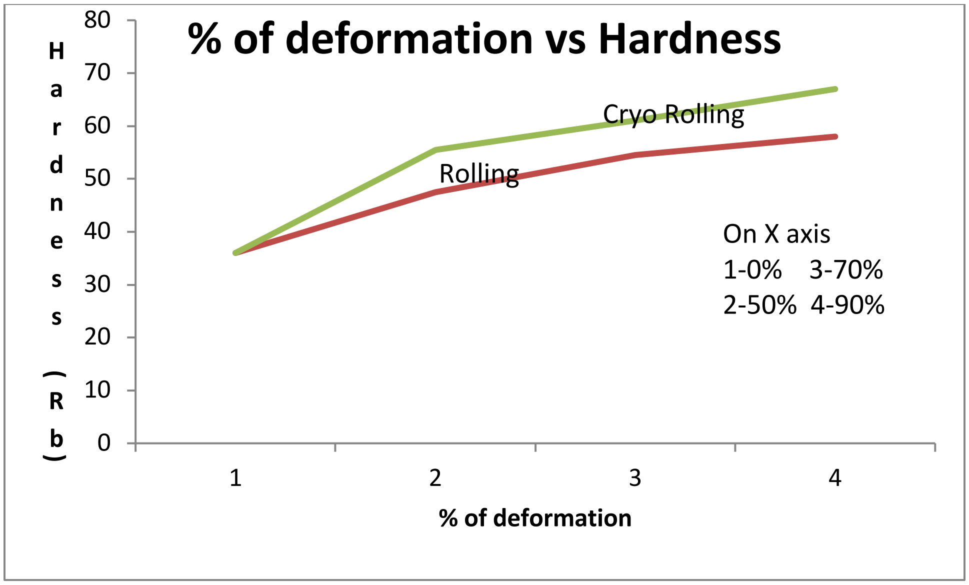
Indentor used: 1/16th steel ball

Table 4.1 : Hardness values of rolled and cryo rolled samples :

Sample	**Hardness (Rb)**
As received	36

Rolled sample	**Hardness (Rb)**
50%	47.5
70%	54.5
90%	58

Cryorolled sample	**Hardness (Rb)**
50%	55.5
70%	61
90%	67



Graph 4.1: % deformation vs Hardness of rolled and cryo rolled samples

Micro hardness values (HV) :

Load : 25g

Dwell time : 10 seconds

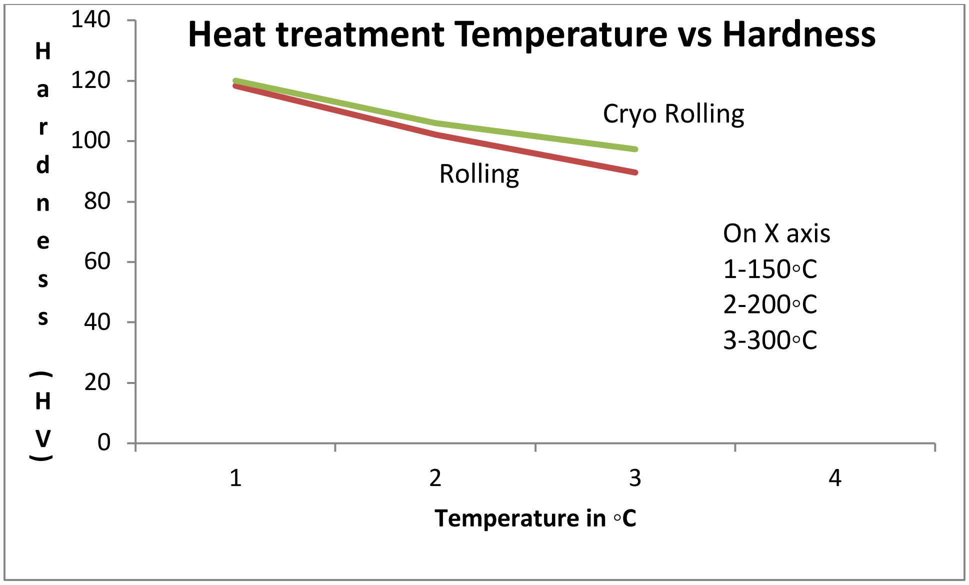
Table 4.2 : Micro hardness values with constant time and varying temperature :

90% rolled:

Temperature in ºC	Time in hours	Hardness (Hv)
150	3	118.3
200	3	102.2
300	3	89.6

90% cryo rolled :

Temperature in ºC	Time in hours	Hardness (Hv)
150	3	120
200	3	105.9
300	3	97.3



Graph 4.2 : Heat treatment temperature vs Hardness

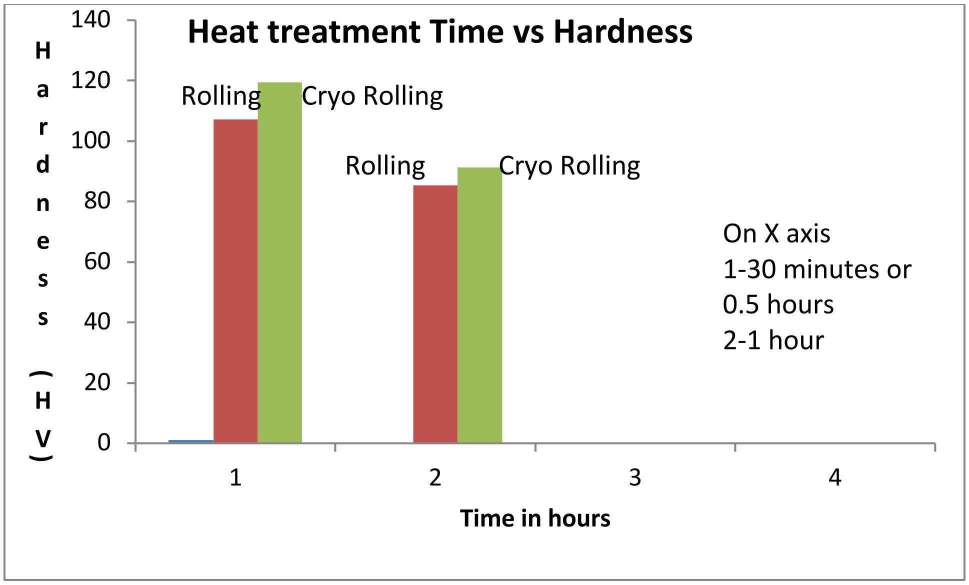
Table 4.3 Micro hardness values with constant temperature and varying time:

90% rolling:

Temperature (°C)	**Time in hours**	**Hardness (Hv)**
300	½	107.2
150	3	85.4

90% cryo rolling:

Temperature (°C)	**Time in hours**	**Hardness (Hv)**
300	½	119.5
300	3	91.3



Graph 4.3 : Heat treatment time vs Hardness

5. Conclusions :

1. The samples with 90% deformation are very difficult to be focused under an optical microscope.
2. It was difficult to distinguish recrystallized microstructure from deformed micro structure under a light optical microscope.
3. Optical photographs showed comparatively more prominent grains in micro structures of the samples heat treated to higher temperatures. Samples heat treated to a temperature of 300^{o}C had more pronounced micro structures than samples heat treated at lower temperatures.
4. SEM scans of rolling planes showed rolled texture.
5. Grains became finer as heat treatment temperature increased to 300^{o}C in rolled samples. They became coarser with increase in time in rolled samples. It was expected that recrystallization would have taken place in rolled samples which is the reason behind the coarsening of grains.
6. However the recrystallization temperature could be obtained with vivid explanation from DSC scans of pure copper.
7. The hardness values of cryo rolling are more than rolling at every phase of the project.
8. Micro hardness values decreased with increasing temperature at constant heat treatment time and decreased with increase in time at constant heat treatment temperature. This indicates superiority of mechanical properties in a cryo rolled sample when compared to rolled sample even after heat treatment.
9. Hardness indentations were more pronounced and clear for 300^{o}C samples.
10. It can be assumed from the results obtained that recrystallisation takes places for rolled samples at 300^{o}C for 30 minutes and for cryorolled samples 300^{o}C for 03 hours but it can be concluded only after DSC characterization of these samples.
11. Optimization and conclusion of recrystallization kinetics and micro structure can be done

thoroughly with characterization of all the samples in EBSD and DSC, and by calculation of grain size and recrystallized fraction. However the experiments conducted with SEM and micro hardness tester could yield results about few assumptions regarding recrystallization.

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