

MANUFACTURING PROCESSES LABORATORY (MSE 315)

Laboratory Manual of 1st set experiments



*Department of Materials Science and Engineering
Indian Institute of Technology Kanpur Kanpur-208016*

2013-14

Instructor
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MANUFACTURING PROCESSES LABORATORY

LIST OF EXPERIMENTS

- 1) A. To study the property changes of ferrous and non ferrous metals during cold and hot rolling
B. Determine the physical changes of the given metals during Swaging
- 2) A. To study the shrinkage behavior during permanent mold casting
B. Molding properties of sodium silicate bonded sand
- 3) Micro-structural Evaluation of MMAW, MAG & TIG Welded Steel
- 4) To Study Various Characteristics of copper Powders and Evaluate Green Density as well as Strength Characteristics (hardness) of Cold-compacted and sintered (Conventional) compact
- 5) To study the behavior copper and ceramic powder during Conventional and Microwave Sintering of Particulate Compacts

MSE315: Manufacturing Processes (2013-14, Sem II)

Laboratory Schedule

Days	Time	Group / Days Distribution & Introduction for whole class on 3/1/2014	Set 1					Lab Quiz 1	Set 2					Lab Quiz 2
			Exer 1	Exer 2	Exer 3	Exer 4	Exer 5		Exer 6	Exer 7	Exer 8	Exer 9	Exer 10	
Monday	9 TO 12		13/1	20/1	27/1	3/2	10/2	24/2	3/3	10/3	24/3	31/3	7/4	10/4
Tuesday			7/1	21/1	28/1	4/2	11/2	25/2	4/3	11/3	25/3	1/4	15/4	
Thursday			9/1	16/1	23/1	30/1	6/2	13/2	27/2	6/3	13/3	27/3	3/4	

First Class : 31st December 2013
 Id-E-Milad : 14th January 2014
 Mid Sem Examination : February 17 – February 22, 2014
 Mid Semester Races : March 15 to March 23, 2014
 Ram Navami : April 8, 2014
 Last Class : April 17, 2014

Important reminders: 1ST TURN: FORMATION OF GROUPS
 HANDLE THE MICROSCOPES WITH CARE
 POLISH AND ETCH EACH SAMPLE CAREFULLY TO GET MICROSTRUCTURE IN SINGLE RUN
 TAKE INITIAL ON THE MEASURED DATA/OBSERVATIONS

Dr. Krishanu Biswas
Instructor In-Charge
kbiswas@, 6184(O)

GENERAL INSTRUCTIONS

1. Every student should obtain a copy of the MSE 315 laboratory manual
2. Dress code: Students must come to the laboratory wearing: (1) trousers, (ii) half-sleeve tops and (iii) Leather shoes. Half pants, loosely hanging garments and slippers are not allowed
3. To avoid any injury, the student must take the permission of the laboratory staffs before handling the machines.
- 4. EVERY STUDENT IS REQUIRED TO HANDLE THE MICROSCOPE/HARDNESS TESTER WITH CARE.**
5. Students must ensure that their work areas are clean.
6. At the end of the experiments, the student must take initials from the staff.
7. Laboratory report must be submitted in standard sheet, available at the shopping center in the subsequent lab turn. Reports on ordinary sheets and computer papers will not be accepted.
8. Each member of any group must submit lab report even if the experiment was performed in a group.
9. The lab report must contain: (i) Title of the experiment, (ii) Three to four lines stating the objectives, (iii) Name of all equipments/tools used along with one line description of its use and (iv) neatly labeled sketch of the observed microstructures with few lines of description.
10. Student can check their laboratory reports after correction.
11. Careless handling of machine, especially optical microscope will not be tolerated.

GENERAL INFORMATION

In this laboratory you will be exposed to various aspect of phase diagrams, microstructure as well as quantitative metallographic techniques. You are required to submit a report on each experiment you will be performing. You will be asked questions on various aspect of the experiments. You need to refer to the text books.

RECOMMENDED READING

- ✓ **Solidification Processing; M.C. Fleming, McGraw-Hill, N.Y., 1974**
- ✓ **Fundamentals of Solidification by W. Kurz, and D.J. Fisher, Trans-Tech Publications, 1989**
- ✓ **Mechanical Metallurgy, George Dieter, MCGRAW-HILL Higher Education, 1989**
- ✓ **Sintering Theory and Practice, Randal M German, John Wiley and Sons, 1996**
- ✓ **Sintering: Densification, Grain Growth and Microstructure, S-J. L.Kang, Butterworth-Heinemann, 2004**
- ✓ **Materials Science of Thin Films, Milton Ohring, Academic Press, 2002**

Experiment No. 1

Deformation Behavior during Rolling and Swaging

INTRODUCTION:

Rolling is the process involving plastic deformation of metals by squeezing action as it passes between a pair of rotating rolls. To control the relative positioning of rolls, a roll positioning system is employed on the mill stand, generally through hydraulic pressure. The most common rolling mill is the 2-high rolling mill, which consists of two rolls usually mounted horizontally in bearings at their ends and vertically above each other rotating in opposite direction. Few pictures of high mills are given in the figure 1.1 The rolls may be driven through couplings at their ends by spindles, which are coupled, to pinions (or gears), which transmit the power from the electric motor. Cross sectional view of arrangement is given in the figure 1.2. The rolling mills could be either reversing or non-reversing type. In the reversing type, which is the most common one, the direction of motion of the rolls can be reversed, and therefore the work can be fed into the mill from both sides by reversing the direction of rotation of rolls. The process may be carried out either at room temperature or at higher temperature. Depending on temperature condition process called cold rolling, warm rolling and hot rolling. Deformation below recrystallization temperature generally $0.3 T_{mp}$ (in Kelvin) is called cold working and deformation above $0.6 T_{mp}$ (in Kelvin) is hot working in between 0.3 to 0.6 will be in warm working category.

- Deformation below recrystallization temperature requires greater force than hot working.
- Above recrystallization temperature plastic deformation causes the deformation of grains and grain boundaries, a general increase in strength, and a decrease in ductility.
- Properties can be brought back to their original levels by heating the piece in a specific temperature range for a specific time at lower temperature known as recovery.

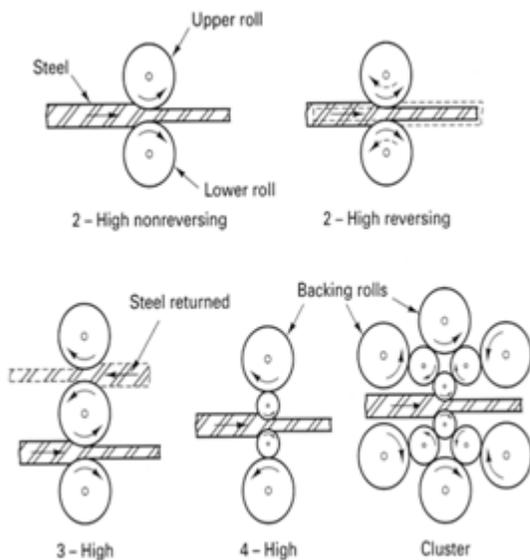


Figure 1.1 Different configuration of rotating mills

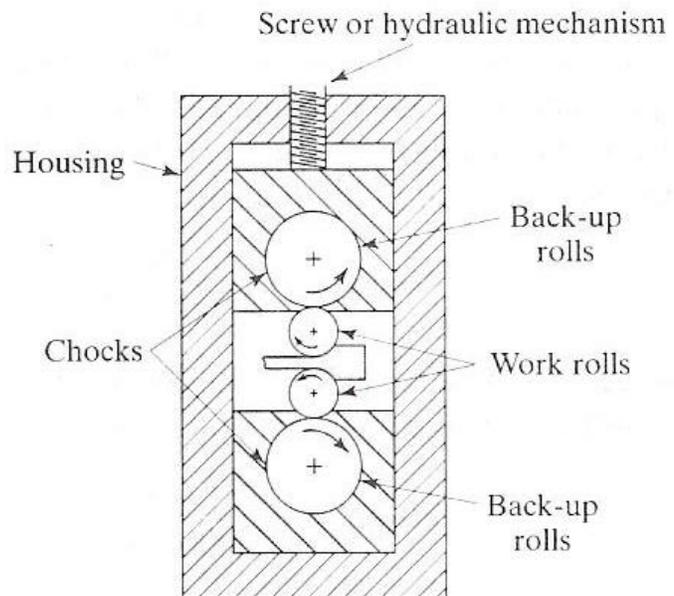


Figure 1.2 Detailed configuration of 4 high mill

OBJECTIVE:

To generate plastic curve of a given metal strip at room temperature and at recrystallization temperature during rolling. Observe the changes in metal characteristic after rolling.

PROCEDURE:

(a) Determination of plastic curve in cold rolling:

- (1) Given metals: Aluminum
- (2) Take 25 -50 mm long various pieces of the given metal strip of the same thickness (~ 5 mm).
- (3) Roll the strips to 10, 20, 30, 40, 50, 60 and 70% thickness reduction is single pass without using any lubricant, and measure the rolling load in each case. Also measure the final thickness in each case (h_a). Tabulate the results.
- (4) Plot a graph between the final thickness after rolling (X - axis) vs rolling load (Y - axis).

(b) Determination of plastic curve in hot rolling:

- (1) Given metals: Mild steel
- (2) Take 25 -50 mm long various pieces of the given metal strip of the same thickness (~ 6 mm) at recrystallization temperature.
- (3) Roll the strips to 5, 10, 15, 20 and 25% thickness reduction is single pass without using any lubricant, and measure the rolling load in each case. Also measure the final thickness in each case (h_a). Tabulate the results.
- (4) Plot a graph between the final thickness after rolling (X - axis) vs rolling load (Y - axis).

OBSERVATION:

Note: Take at least 5 readings in each case.

(1) Data set No.(a) for plastic curve:

Rolling condition: No lubricant used

S. No.	Roll gap, mm	Initial thickness of strip, mm	Final thickness of strip, mm	Rolling load, Ton
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(2) Data set No.(b) for plastic curve:

Rolling condition: No lubricant used

S. No.	Roll gap, mm	Initial thickness of strip, mm	Final thickness of strip, mm	Rolling load, Ton
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If time permits observe microstructural changes during rolling.

CALCULATION:

Plot the following two set of graphs.

- (i) The final thickness of the metal strip (X-axis) vs rolling load (Y-axis) for both the cases (cold and hot), i.e. plastic course, on the same graph.
- (iii) Superimpose the curves obtained from the both data on the same graph.
- (iv) Report the hardness changes.
- (v) Report the microstructural changes.

QUESTIONS:

- (1) Differentiate the hot and cold rolling and what are the effects on metals during both rolling processes?
- (2) What is the utility of backup rollers?
- (3) Explain the phenomena of spring back in rolling?
- (4) Explain the metallographic changes after rolling?

Swaging:

Swaging is a forging process in which a solid rod is subjected to radial impact forces by a set of reciprocating dies. The die movements are obtained by means of a set of rollers in a cage, an action similar to roller bearing. The work piece is stationary and the dies rotate, striking the work piece at rates as high as 20 strokes per second. The forming by swaging converts kinetic energy into impact energy and then into forming forces, applied by the die to the work piece. The workpiece is fed lengthwise into the die and the reduced end will increase the length during the forming process. This process is used to reducing the regular or irregular cross-section area of tube, produce a taper, or add point to a round work piece. In tube swaging the internal diameter of the hollow work pieces can be controlled with or without the use of internal mandrels (see Figure 2.1). The small-diameter tubing, high strength wire is used as a mandrel. Mandrels can also be made with longitudinal grooves, to allow swaging of internally shaped tubes. For example, the rifling in gun barrel is made of tube swaged over a mandrel with spiral shaped grooves.

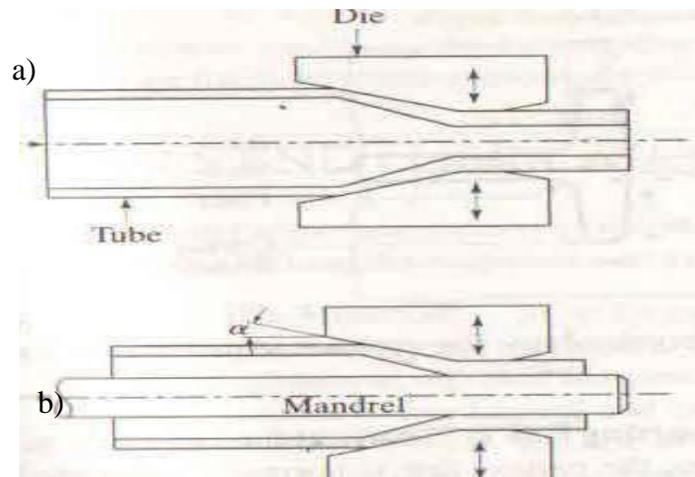


Figure 2.1 Swaging operation on a tube a) with and b) without mandrel

Typically swaging is performed at room temperature and like any other cold working process, swaged parts show improved mechanical properties. However, for workpieces having low ductility at room temperature, swaging operation is also performed at higher temperatures. Lubricants are used to give good surface finish of the work piece and to prolong the die and mandrel life.

OBJECTIVE:

1. To demonstrate the swaging behavior of a given rod .
2. To measure the reduction in area, Rockwell hardness and the microstructure of the swaged pieces.

PROCEDURE:

1. Swage the given piece to different amounts of area reduction.
2. Measure the hardness after reduction.
3. Present your data in the form of a graph between (i) hardness vs engineering strain and (ii) hardness vs true strain.
4. Observe the microstructure after reduction.

REPORT:

1. Objective of the experiment.
2. Report the strain, hardness and microstructure after reduction pass.
3. Report the microstructure after reduction.

S.No	Die Size	Initial Diameter	Final Diameter	Strain	Hardness	Remarks on Microstructure
1						
2						
3						
4						

QUESTIONS:

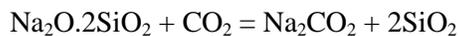
1. What type of forces developed during swaging?
2. Explain the unique aspect of swaging?
3. Discuss the possible swaging defects & how does swaging influence the mechanical properties?

Experiment-2

MOLDING BEHAVIOUR OF SODIUM SILICATE BONDED MOLDING SAND

Introduction:

Though green and dry molding sands containing clay as the binder are commonly used in the foundry practice, binders other than clay such as sodium silicate, molasses, linseed oil etc. are also frequently used as binders. Mold and core making processes based upon the sodium silicate represent a high proportion of the total chemically hardened sands used in the foundry industry. Advantages of using sodium silicate are that (i) it is cheap and easily available, (ii) it is inorganic and does not evolve gases upon heating when the hot liquid metal is poured, and (iii) it does not collapse and burns out after casting and hence gives thermally stable sand. However, in order to generate a proper bond it is necessary to treat the sand mixture with CO₂ gas (hence the process is also known as the CO₂ process). The chemical reactions between sodium silicate and CO₂ are:



In the reactions SiO₂ separates as the silica, which causes an increase in the viscosity of the binder and hence an increase in the strength of the bond. In addition to these chemical reactions, some loss of water from the silicate occurs when the CO₂ is passed, which also causes an increase in the viscosity of the bond and increase in its strength. Thus if a proper bonding has to be achieved, the gassing time and the pressure are to be controlled properly.

Objective:

To study the technique of preparing sodium silicate bonded sands and to test their bonding properties.

Procedure:

1. Prepare adequate quantity of sand mixture containing 2% of bentonite clay, 1.5% water and a known % of sodium silicate. For this purpose (i) charge the sand and water in the Muller and mull the mixture for 2-5 min, (ii) stop the Muller and sprinkle the bentonite clay and mull for 2-5 minutes and (iii) stop the Muller and slowly add the sodium silicate and again mull for 4-5 minutes. Collect the mixture thus prepared in a polythene bag.
2. Prepare standard figure of eight specimen using a sand rammer. Estimate the weight required to obtain a sample of 1" height by making a trial sample first. Make 3 samples first and put them in the oven maintained at 150°C for drying for 40 minutes. After achieving drying in this way take them out from the oven and cool them in air for 15 minutes.
3. Make 3 more samples and gas each sample with CO₂ while the samples remain in the sample holder mold (gassing pressure to be kept as 0.3 kg/cm³).
4. Keep the gassed sample for aging for 1 hour in air.
5. While the samples prepared in Step-4 above are undergoing their aging, prepare 3 more samples by gassing and test them immediately without aging for their strength and hardness.
6. Test the dry samples as well as aged samples for their strength and hardness.

Report:

1. Title of the experiment and its objective
2. Ingredients in the sand mixture, method of its preparation, gas pressure and reaction time, aging time.
3. Schematic diagram of the gassing operation.
4. Tabulated results of moisture contents, strength, permeability and hardness of variously treated samples.

Questions:

1. Compare the green sand and sodium silicate bonded sand
2. What is the effect of aging time on the bond strength of the gassed sodium silicate bond?
3. What are the disadvantages of the sodium silicate process?

Casting Defects in Materials Cast in Permanent Molds

INTRODUCTION:

When a liquid metal is poured in a stationary metal mould, and allowed to solidify, pipe formation inside the solidified ingot occurs. Pipe is the shrinkage cavity formed in the ingot. Pipe is of two types – Primary and Secondary. Primary pipe is located in the upper central portion of the ingot. The Secondary Pipe is located inside the ingot. The ingot mould is usually tapered from the top to the bottom of the mold, primarily to facilitate stripping of the ingot after solidification. The taper shape gives rise to the two principal types of moulds – big – end up and big – end down. The rate at which heat is extracted from an ingot solidifying in a mould, and hence to rate of solidification, is affected by many factors, some of which are the thickness, shape, and temperature of the mould, the superheat of the liquid metal, and the cross-section of the ingot. The nature of pipe formed in ingots greatly depends on the type of mould used. In the present experiment, the effect of big – end up and big – end down type moulds on the pipe formation will be studied.

OBJECTIVE:

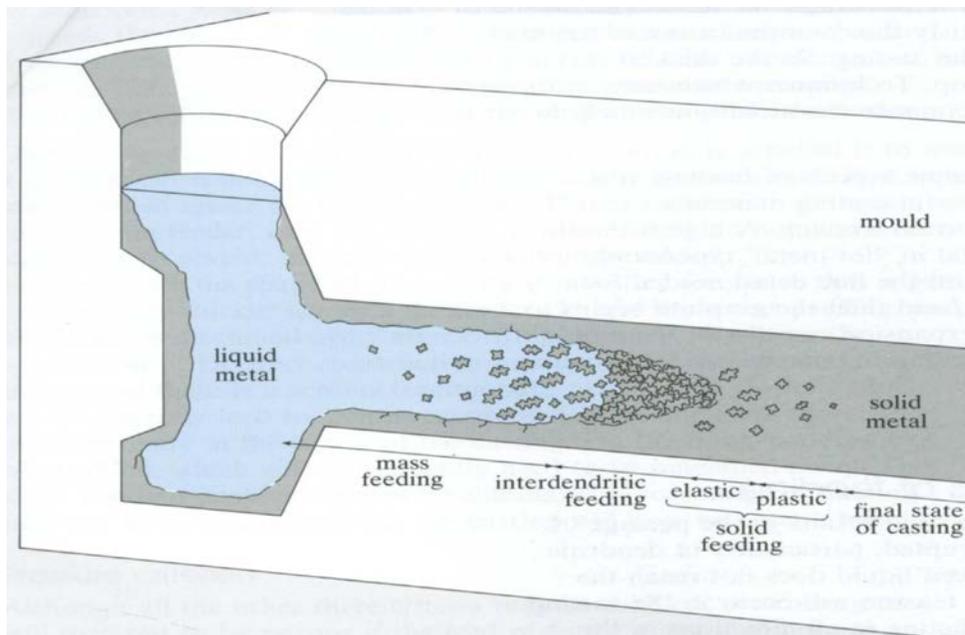
To study the pipe formation in a cast metal ingot.

PROCEDURE:

1. Melt the given metal/alloy.
2. Pour the liquid metal/alloy in ingot shape mould made from steel. Use two casting conditions – Big- end up and Big-end down. Allow it to solidify.
3. Measure the volume of the primary pipe formed in the upper portion of the ingot.
4. Cut the solidified ingot in the longitudinal direction.
5. Measure the volume of secondary pipe formed inside the ingot.
6. Calculate the yield of the sound metal in the ingot.

REPORT:

1. Objective of the experiment
2. Materials used
3. Procedure in your words, including the melting of metal/alloy.
4. The volume of primary and secondary pipe.
5. Yield of sound metal in %.
6. Schematic diagram of the moulds
7. Schematic diagram of the cross – section of the ingot showing the pipes
8. Draw a typical ingot structure showing the casting defect for killed steel in the following four cases –
 - a) Big – end up, hot topped
 - b) Big – end down, hot topped
 - c) Big – end up, without hot topped
 - d) Big – end down, without hot topped.



Experiment – 3

Microstructural Evaluation of MMAW, MAG & TIG Welded Steel

INTRODUCTION:

Fusion welding methods are the most effective ways for joining metals and alloys. These methods play a very important role in the manufacturing industries. They are employed in manufacturing of products such as ships, railways, earth moving equipments, automobiles, space vehicles, buildings, bridges, farm equipments mining equipments, furnaces, boilers, home appliances and many other products of common use. These processes include oxy-fuel gas welding, arc welding, thermit welding, electro-slag welding, electron beam welding, laser welding etc. Arc welding is the most common fusion welding methods for joining ferrous as well as non-ferrous metals and their alloys. Shielded metal arc welding, gas tungsten arc welding, gas metal arc welding, flux core arc welding, submerged arc welding, plasma arc welding and electron gas welding are the fusion welding processes belonging to the family of the arc welding processes. Fusion welding processes, however, are among some of the most complex metallurgical processes since large variety of metallurgical phenomena occur in a brief time interval when the weld bead is being made. The engineer, when specifying the welding operation, is concerned with gas-metal reactions, slag-metals reactions, solidification and heat-flow considerations, solid-state reactions and transformations, and of course reliability and cost. Thus the engineer is not just concerned with the area of coalescence produced by the fusion welding, but with the entire welded joint which includes the region around the coalesced zone as well as the zone itself.

The manual metal arc welding (MMAW) is the most basic form of arc welding. It uses high current and low voltage electricity to form an electrical arc. It has a consumable electrode, which conducts the arc onto the work metal, melts in the process and forms filler metal. The arc must be manually started; i.e, by striking the workpiece with the electrode like lighting a match. All metals react with oxygen in the air, and react much quicker when temperatures are elevated. MMAW overcomes this by including a coating on the electrode that vaporizes along with the metal and forms a cloud of slag that keeps the oxygen out. This slag solidifies on the weld and is chipped away to see the weld. The metal active gas welding (MAG) is a sophisticated form of welding. It works on the same principle as MMAW with the different ways of dealing with shielding and electrodes. It uses a continuous wire as an electrode. The wire is continuously fed, at the rate at which it is used up into filler metal. It uses CO₂ to shield the working area and prevent oxidation of welded metal part. This means that one can weld more reactive metals, such as aluminum. There is no slag to be chipped away in MAG welding.

A designer must anticipate two problems inherent in the fusion welding process: (1) the effect of localized heating and cooling on the microstructure and properties of the base metal and (2) The effect of the residual stresses that are locked in the weldment as a result of the uneven cooling of the weld deposit. The heat affected zone (HAZ) is the region of the base metal, adjacent to the weld bead, where the temperature has caused the microstructure of the base metal to change. In carbon and alloy

steels HAZ is particularly important because of the phase changes that occur in them when they are subjected to heating and cooling from high temperature. The depth of penetration in fusion welding is affected by the welding velocity, welding current, and the degree of preheat. It influences the preparation of the joint and the number of phases required to complete the weld. For ferrous materials peak temperature and cooling rate are important factors from the point of view of predicting whether rates are important for predicting whether the martensitic transformation will occur or not.

OBJECTIVE:

To study the effect of heat affected zone on the microstructure of steel weldment.

PROCEDURE:

1. Take pieces of 5 mm thick, 25 mm wide and 80 mm long steel plates and remove dust/rust/oil from the surfaces them using a sand paper or cloth. Weld the two pieces of steel plates by MMAW process, MAG Process and TIG Process as described below.
2. Heat input to the job is related to the welding current. Welding current is changed by varying the open circuit voltage of the welding machine and the slope of the volt-ampere curve. Set the desired welding current.
3. Lay a weld bead at the centre of the plate at an average speed of about 10 mm/min by striking the arc and moving the electrode manually till about 20 mm long bead is formed.
4. When the welded plate is cooled to a temperature $< 500^{\circ}\text{C}$, remove the plate from the table and cool it to room temperature in running water.
5. Remove the slag if deposit from the welded plate by wire brushing. A clean bead will be visible.
6. Cut all the welded plates vertically so that the bead cross-section can be examined and polish the sectioned surface for its visual and micro structural examination.
10. Measure the size of the pool and the depth of penetration.
11. Measure the hardness of the welded joint as a function from the distance from weld axis.
12. Etch the polished surface and observe the microstructures of various weld zones. Estimate the width of the heat affected zone (HAZ) in each case.

REPORT:

1. Objective of the experiment.
2. Appearance, pool size and depth of penetration as a function of different processes.
3. Hardness vs. distance plots for various processes.
4. Size of the heat affected zone (HAZ) as a function of welding processes.
5. Microstructures and associated differences with respect to different welding processes.
6. Hardness profile of weld joint as a function of distance from the weld axis.

EXPERIMENT – 4

To Study Various Characteristics of given metal powders and Evaluate Green Density as well as Strength Characteristics (hardness) of Cold-compacted and sintered (Conventional) powder

INTRODUCTION

Powder metallurgical (P/M) processing of the metals and alloys plays an important role in manufacturing various engineering components for several applications. The conventional P/M technology for making components starts with metal powders as the raw material, which is pressed in dies of suitable shape to produce green compacts. These green compacts are subsequently, sintered at high temperature under protective atmosphere with following objective: (i) to develop proper bonds between higher to mechanically pressed powder particles comprising the green compacts and (ii) to reduce the porosity level in the sintered compact so that it has still higher relative density. However, parts produced by the conventional P/M approach always contain some residual porosity. Non-conventional P/M technologies have been developed with the objective of completely eliminating the porosity from the manufactured part.

Metal powders are produced by various methods, such as (a) atomization, (b) solid-state reduction of metal oxides or other compounds, (c) chemical precipitation from organic or inorganic solutions, (d) electrolysis, (e) mechanical alloying etc. Each of these generic processes may have several specific variants for metal powder preparation. The shape, size and their distribution of as-synthesized metal powders depend on the specific method of production, variables associated with the process and their control. The tapping of a powder stock in the die and its behavior during compaction and sintering is substantially influenced by its size and shape characteristics. It can be mentioned here that the spherical powder particles with uniform size distribution is usually preferred from the point of view of good flowability during compaction as well as far good sinterability.

Metal powders have different shapes such as spherical, rebounded, dendritic, acicular, rod-like flaky, irregular etc. An analysis of size and its distribution of a given batch of powder, coarser than 44 μm , are generally carried out by sieves. There are specialized methods for determining the size and its distribution for powders, finer than 44 μm , i.e. the powder of sub-sieve size.

The density of loose powder is known as the apparent density. It is measured by allowing the loose powder to flow through the Hall flow meter. Very often, the loose powder is mechanically vibrated and/or tapped so that the particles in the loose mass further settle and occupy a smaller volume. The density of the powder mass obtained after its mechanical vibration or tapping, is known as the tap density. The apparent density and tap density are useful indices for understanding the behavior of the powder during its compaction.

It has already been discussed in class lectures that for most of the conventional powder metallurgy applications for manufacturing various engineering components, the metal powder has to be compacted in dies of the appropriate shape. Compressibility of a given metal powder depends on various physical properties of the metal or alloy and also on the (a) size and shape of the powder and (b) friction characteristics and the die/powder interface. Thus the compressibility of a metal powder is an index of its densification behavior under the application of an external pressure. The compressibility of a powder compact is often measured in terms of the green density, achieved at a given applied pressure. Typical illustration of the cold compaction process to obtain a green powder compact is shown in Fig. 4.2. Alternatively, it is defined in terms of a densification parameter, which is given as,

$$\text{Densification parameter} = \frac{\text{Green Density} - \text{Apparent Density}}{\text{Theoretical Density} - \text{Apparent Density}}$$

From the above definition of the densification parameter, it is clear that it represents the fractional densification achieved with respect to the theoretically maximum attainable densification. Green strength of a compact is expressed as Transverse Rupture Strength. The schematic illustration of the three point flexural set up is shown in Fig. 4.3. In the transverse rupture strength test, specimens are broken in a special testing fixture. The specimen in the testing fixture is supported by two hardened steel rods. Another rod presses the specimen at the centre till it breaks. The green strength of the powder compact is calculated from the following relationship.

$$S = \frac{3PL}{2t^2W}$$

where,

- S = Transverse rupture strength, kg/m²
- P = Breaking load, Kg.
- L = Distance between the supporting rods, mm
- t = Thickness of the green specimen, mm
- W = Width of the green specimen, mm

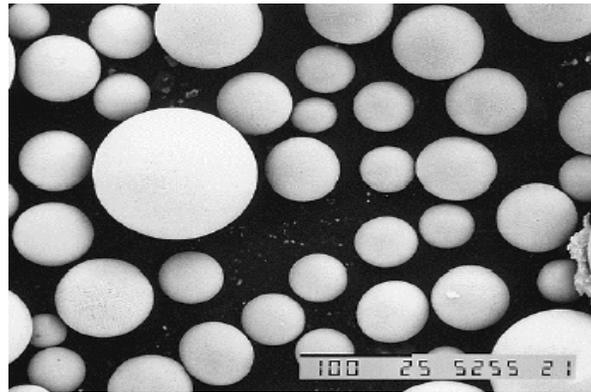


Figure 4.1: Scanning electron microscope image revealing typical size and shape of Nickel-based superalloy powder particles synthesized by the rotating electrode process.

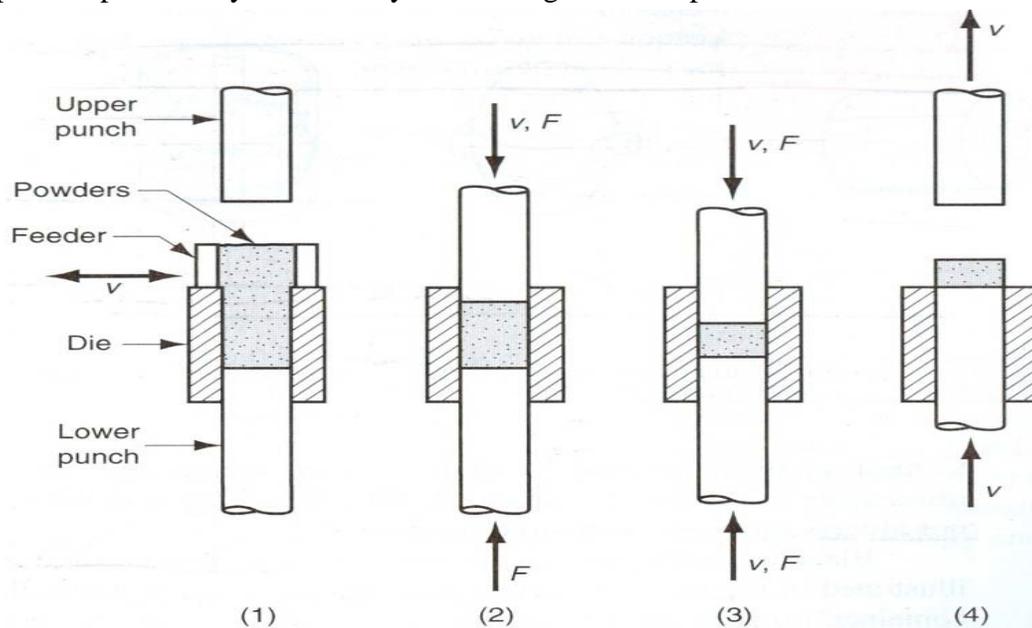


Figure 4.2: Conventional method of compaction: (1) Filling die cavity with powder by automatic feeding system, (2) initial and (3) final positions of upper/lower punches and (4) ejection of sample.

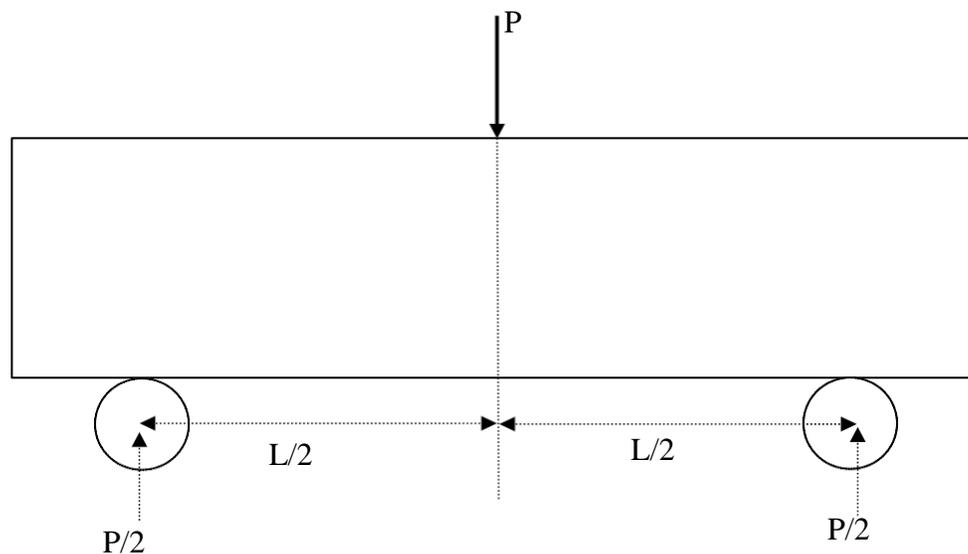


Figure 4.3: Schematic illustration of three point flexural strength measurement of rectangular cross section bar.

OBJECTIVES:

1. To study the particle characteristics of the given metal powders.
2. To study the die compaction behavior of the given metal powder and to determine the green density and the green strength of the green compacts.

PROCEDURES:

A. Powder Size and Characteristic

1. Weigh about 100 g of metal powder given to you.
2. Sieve the weight powder in the set of sieves for about 20 minutes
3. Weight the powder on each sieve and tabulate the weights as weight percentages of the whole mass.
4. Observe different batches of powders under the stereo-microscope.
5. Neatly sketch the powder particle morphology of powders given to you for the observation of their shapes.

B. Powder Density

1. Taking about 150 – 200 g of the given powders and gradually pass them separately in the graduated measuring cylinder of the Tap Density Meter.
2. Gradually make the upper powder level horizontal and measure the volume. Calculate the tap density of both the batches of given metal powders.
3. Introduce vibrations in the powder by taping (5 at a time) and observe the decrease in powder volume. Calculate the density of the powder. Repeat this procedure till the density approaches a stable value.

C. Green Density

1. Weigh pre-determined mass of the powder for preparing the rectangular green compact.
2. Compact these powder at three different loads. Calculate the applied pressure at the three different loads.
3. Eject the green compact from the die carefully and weigh each green compact.
4. Measure the dimensions of green compacts and calculate the densities of the compacts followed by volume.

D. Green Strength

1. Measure the width and thickness of the green compact in mm.
2. Carefully place the rectangular green compact over the fixture. Take care that the specimen projects equally on both the sides of the fixtures.
3. Switch on the loading device (it automatically stops once the specimen breaks). Read the breaking load.
4. Calculate the green strength of the entire three specimens.

REPORT:

1. Objective of the experiment.
2. Particle size distribution of the given powder in terms of the particle size (in sieve size as well as in microns) distribution histogram.
3. Cumulative particle size distribution of the given powder.
4. Morphologies of the given powders indicating the presence of features such as, powder shape, size, distribution etc.
5. Apparent density of powders.
6. Tap density of the powders as a function of tapping vibrations and the effect of powder shape and morphology on the apparent density and the tap density of the powder.
7. The basic features of the powder compaction press.
8. The basic features of the green strength testing device.
9. Plot the green density values with compaction pressure.
10. Plot the green strength of powder compacts with compaction pressure.

QUESTIONS:

1. What are the limitations of your sieve shaker? What improvements can be made?
2. What will be the effect of various particle shapes and morphologies on the results obtained from the sieve analysis? Why is it difficult to sieve metal powder particles finer than 325 mesh size?
3. How would be the accuracy of the tap density data affected by the volume of the metal powder taken for the sampling?
4. What was the production methods of the given metal powder used in your experiments? If you were given a powder produced by a different method how do you expect your results to be different from what you have got.
5. How is particle size expected to affect green strength and density variations with pressure?
6. How does the compaction pressure affect the density of the powder compact prepared from a (i) soft and (ii) hard powder of the same material?

Experiment – 5

Conventional and Microwave Sintering of Particulate Compacts

Sintering refers to the process of firing and consolidation of powders at $T > 0.5T_m$, where diffusional mass transport leads to the formation of a dense body. It is a powder metallurgical based technique to produce high density materials and components from metal or ceramic powders by applying thermal energy and/or mechanical pressure.

A large number of metallic engineering components are manufactured on a commercial scale by sintering the compact prepared from the chosen metal powders. Sintering can be classified as Solid State Sintering and Liquid Phase Sintering. In solid state sintering, all the constituents in the powder compact are in the solid state during sintering.

Solid state sintering is the process by which particles bonded to each other at temperatures below the melting point by atomic transport events. The driving force for sintering is a reduction in the system free energy, manifested by decreased surface curvatures and an elimination of surface area. The driving force provides the stress on the material that leads to mass flow; this is usually the surface energy. The surface energy per unit volume depends on the inverse of the particle diameter. Thus finer particle size increases the driving force.

The sintering mechanism describes the path of atomic motion which produces the mass flow. The transport mechanisms are the ways in which mass flow occurs in response to the driving forces. There are two classes of transport mechanisms – surface transport and bulk transport. Surface diffusion and evaporation – condensation are the two most important contributors during surface transport controlled sintering. The bulk transport mechanisms include volume diffusion, grain boundary diffusion, plastic flow, and viscous flow. Plastic flow is thought to be of minor importance. This is shown schematically in Fig. 8.1b. The changes in pore size and shape are also demonstrated in Fig. 8.1a.

The stages of sintering describe the geometric progression associated with the sintering process. There are three stages in sintering. During the initial stage sintering, the particles form bonds with rapid neck growth. Local curvature gradients in the neck region provide the initial driving force. With prolonged sintering, the pore structure becomes smoothed, eliminating the major curvature gradients, leading to the intermediate stage. The intermediate stage represents an open, continuous pore structure. The driving force for mass flow is the interfacial energy associated with the pore surface. In the final stage of sintering, grain growth occurs along with pore shrinkage during the later portion of sintering. As a result of it, the pores become spherical and isolated. Elimination of isolated pores is difficult.

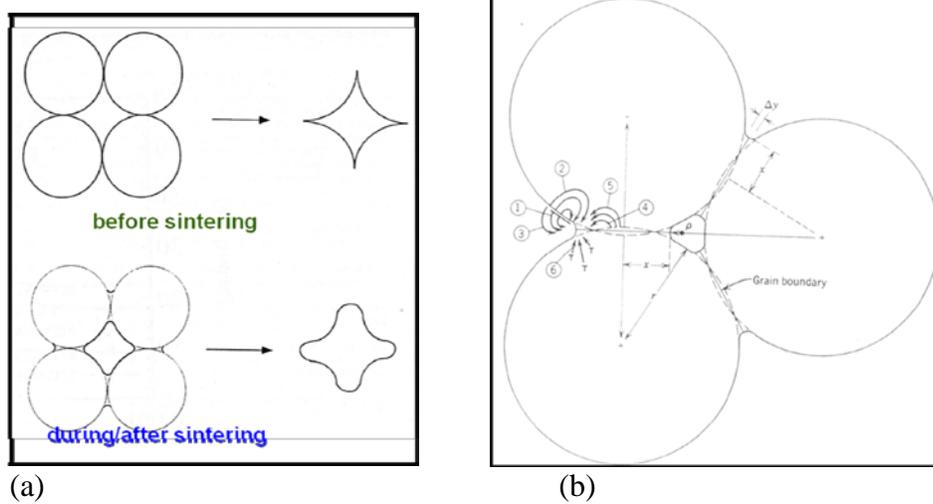


Fig. 5.1: Schematic illustration showing, (a) changes in pore size and shape during sintering, (b) different mass transport phenomenon involved in neck growth during sintering

COMPACTION EFFECTS ON SINTERING:

The higher compaction pressures contribute to an increase in density and neck contact size in green compact, while reducing the rate of neck growth during sintering. Further, the higher compaction pressures lead to larger net neck sizes. Also, the shrinkage decreases with an increasing compaction pressure. It is the sintered neck size which dominates the properties such as strength and ductility, thus, higher compaction pressures are generally beneficial. Increasing the compaction pressure gives better dimensional control, less sintering shrinkage, and better final properties.

OBJECTIVES:

- a) To study the sintering behavior of a powder compact prepared from copper powder.
- b) To measure the green strength and hardness value of the sintered powder compact.

PROCEDURE:

1. Blend copper powder and 0.5 wt. % zinc stearate lubricant.
2. Prepare cylindrical compacts from the blended powder mixture at three different pressures using the given die and punch. Also, apply the lubricant on the walls of die and punch. Select the pressure in such a way that there is no problem in handling the compacts.
3. Weigh the compacts and measure their dimensions, i.e. diameter and thickness, and calculate the green density of the pellets.
4. Place the green compacts over a Nimonic tray and insert the assembly in the hot zone of the tubular furnace, kept at the selected temperature of 850°C for 30 minutes. Close the furnace ends and introduce a protective gas inside the furnace.
5. Allow a sintering time of 30 minutes for all the compacts. Switch-off the furnace and take out the Nimonic tray gradually from the hot zone. Cool it in the cold zone for sufficient period of time.

6. Measure the dimensions of the sintered compacts and weigh them again. Calculate the density of the sintered compacts.
7. To chemically etch the samples and observe the microstructure
8. Also, measure the hardness values of each sintered compacts.

REPORT:

1. Objective of the experiment.
2. Starting powders together with the size distribution of the copper powder.
3. Procedure adopted in your words.
4. Observations i.e. dimensions before and after sintering in a tabular form.

Sr. No.	Compaction Pressure, MPa	Green Compacts			Sintered Compacts		
		Wt, g	Dia, mm	Thickness, mm	Wt, g	Dia. mm	Thickness, mm
1							
2							

6. Calculation: Calculate green compact density, sintered compact density, sintered compact porosity and the densification parameter. Use the following formula for calculating the densification parameter. Take the theoretical density of copper as 8.94 g/c.c & Copper oxide as 6.31 g/cc . Report in a tabular form.

Sr	Compaction Pressure MPa	Green Compact Density g/cc	Sintered Compact Density g/cc in Conventional	Sintered Compact Density in Microwave	Densification Parameter

7. Plot the variation of green density, sintered density, sintered porosity and the densification parameter with the compaction pressure.
8. Plot the Time vs temperature of the microwave sintering furnace.
9. Report the microstructure and hardness values
10. Write a brief comment on your result.

QUESTIONS:

1. In what way, the industrial sintering furnace is different than that used by you for this experiment? Draw a neat sketch of a typical industrial sintering furnace showing different zones together with its position – temperature profile.
2. A highly oxidized copper powder was used for making a compact having 80% green density. What problems would be faced during the sintering of such a compact?
3. What is the effect of particle size on the sintering of metal powder?
4. What is loose powder sintering? State its applications.

