EXPERIMENT NO 9

Microstructural characterization and phase analysis of sintered products

Objective:

Characterization of sintered alumina compact

Basic Theory:

When a green compact is sintered, the original particle boundaries can no longer be observed. Instead, the structure becomes similar to that of the metal in wrought and annealed conditions, except that it contains pores.

Pores are of two types:

- Open
- closed

With progression of sintering, pores continue to shrink.

- At about 5% total porosity, the formation of closed pores commences.
- Many of the microstructures seen in P/M parts are caused by porosity and by the blends of elemental powders that constitute many alloys.
- These blends do not always result in homogeneous, well-diffused structures.

In general, the density achieved in sintered products is between 70 and 95 % of the fully dense wrought products, depending on the production technology in use and the type of application.

Pores are of two types:

(a) interconnected

(b) closed or isolated

- In the first case the pores are connected with each other along the particle junctions. The pores are consequently irregular, unless the particles are initially spherical. Such pores can remain as low as 5% of total porosity.
- The latter types of pores, i.e. closed pores, are pronounced when total porosity is low (< 5%). They are often, but not necessarily, spherical.

X Ray Diffraction (XRD)

- X Ray Diffraction (XRD) is used to obtain structural information on an atomic scale from both crystalline and non-crystalline (amorphous) materials.
- XRD is a non-destructive technique and can be successfully applied to determine crystal structures of various types of materials such as metals, ceramics, alloys and inorganic compounds, in both the thin film/coating and bulk forms.
- XRD also can be applied to obtain structural information such as lattice strain, crystallite size and crystal orientation.

- The X-ray diffractometer is a versatile instrument for phase and structural analysis of metals, thin-films and powders.

Structure is general term, which is used to cover a wide range of structural features from visible to the naked eye down to those corresponding to the inter-atomic distances in the crystal lattice. Structure is classified into four categories: macrostructure, mesostructure, microstructure and nanostructure.

Microstructural analysis covers two aspects:

- Qualitative: The qualitative includes the examination of various phases and their distribution.
- Quantitative. In quantitative metallography, the measurements of the grain sizes and amounts of various phases are established.

Polished cross-sections of a powder compact provide information on the grain and pore structures.

- Scanning electron microscopy (SEM) is useful in visualizing three- dimensional nature of pores.
- The SEM becomes the most important analytical tool in powder metallurgy.
- Electron microscopy provides an overview of the microstructure.
- Most compact properties are related to the array of features measurable via metallography.
- The compact processing and initial powder characteristics will dictate microstructural features.
- Hence, close examination will reveal the processing history and probable properties of a powder compact.
- The standard features such as inclusions, oxide particles, second phases, and phase transformation products are observable by metallographic analysis.

Equipment/ Raw Materials:

The raw materials consist of

- Sintered alumina compact
- X-Ray Diffractometer (Panalytical X'Pert PRO)
- Field Emission Scanning Electron Microscope
- Pin Stubs
- Carbon Tape

Procedure:

Metallographic sample preparation

1. Remove approximately, 0.2mm of the surface of compacts by grinding on a belt grinder
2. Polish the sample surfaces to mirror finish using abrasive papers
3. Final polishing is done on a flocked rayon cloth using colloidal alumina solution
4. Clean the surface using water or with ethanol
5. Clean the surface using water or with ethanol and dry carefully

Density measurement

The sintered samples were ground, polished, thoroughly cleaned in acetone medium and then dried. The weight of cleaned sample is taken using an electronic digital balance. The density of the sintered pellet is measured using Archimedes principle with water as the immersing medium. Taking the ratio of weight in air to the volume of dispersed water carried out the density measurements. Let the weight of a sample in air is W_a, and that obtained by immersing in a fluid be W_f. So, the apparent weight loss of the sample is (W_a-W_f). We can say that the weight of the dispersed fluid is (W_a-W_f). If the fluid has a density of D_f, then the volume of the displaced fluid is

$$\left(\frac{W_a - W_f}{D_f}\right) \quad (1)$$

which is equivalent to the volume of the sample. Therefore,

$$\text{Density of the sample } (D_c) = W_a \Big/ \left(\frac{W_a - W_f}{D_f}\right) \quad (2)$$

For water to be the immersing medium, $D_f = 1$ g/cc, and hence the density of the sample would be

$$D_c = \left(\frac{W_a}{W_a - W_f}\right) \quad (3)$$

X Ray diffraction (XRD) study

- X-ray diffraction studies are carried out for the purpose of phase identification of the sintered samples of Al_2O_3.
- This study is made by using Panalytical X'Pert PRO diffractometer operated with Cu-K_α radiation (having wavelength=1.54 A) on X-ray diffractometer.
- The X-ray source is operated at a current of 20 mA and a voltage of 40kV.
- The diffraction angle is varied in the range of 10-100^0.
- The range of 2θ is selected such that all the major peaks of the expected phases to be present in the powder are covered.
- Subsequent peak fitting is performed in Xpert HighScore software, wherein the peaks were analyzed for the presence of different phases.

Scanning electron microscopy study

Microstructural investigation of the polished samples is performed using secondary (SE) electron imaging mode on a Field Emission Scanning Electron microscope (FESEM) to examine the distribution of the constituent phases.

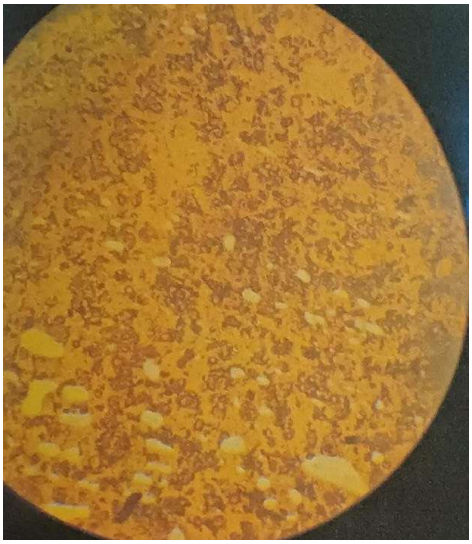
Lab Deliverables

1. Determine density of the sintered compact.
2. Index XRD pattern of sintered alumina compact
3. Describe microstructural observation of sintered alumina compact and list the microstructural features that are observed in the current laboratory experiment.

Observations and calculations:

Magnification = 200X

Microstructure of Cu-10% Sn:



Microstructure of Cu-30%Sn: