CHAPTER 4: EXPERIMENTAL DETAILS

4.1. Introduction

In this chapter, the various procedures followed during the experimental work are described in detail. The experimental work is aimed at the investigations on the workability and mechanical properties of the sintered low alloy steels under investigation.

4.2. Raw materials

The following elemental metal powders were employed for the present research work:

- Atomized iron (Fe) powders of particle size of 150 µm and purity 99.5% supplied by M/s Hoganas India Ltd., Pune, India.
- Graphite powders of particle size 5 µm supplied by M/s Ausbury Graphite Mills, USA.
- Nickel (Ni) powders of particle size 100µm and purity 99.5%, Chromium (Cr) powders of particle size 100µm and purity 99% and Molybdenum (Mo) powder of particle size 100µm and purity 99.55%.

4.3. Mixing

The required mass of Fe, graphite, Cr, Ni and Mo powders were accurately weighed and mixed in a pot mill for 10 hours at 85 rpm, in order to achieve homogenization of the powder mix.
4.4. Compaction

Die compaction is the most common means for densifying a powder and shaping it to the required preform. Compaction of powder is done by using die and punch. During die compaction, pressure is applied through the upper and lower punches to press the powder into the desired shape. The die compaction cycle consists of four steps; fill position, pressing position, compaction, and finally ejection. Compaction cycles are categorized as single, double, and multiple actions. Single action compaction was used in this study.

The blended powder mass was then compacted into cylindrical billets of 24 mm diameter and 12 mm height using a hydraulic press of 1000 kN capacity, applying the required pressure for achieving a sintered density of 83 ± 2% of theoretical density for upsetting and cold repressing studies. The corresponding pressure required was obtained through compressibility tests. Compacts of 24 mm diameter and 32 mm height with sintered density of the order of 95 ± 1% of theoretical density were produced from the same powder mix for preparing cylindrical tensile and square impact test specimens. Graphite powder- lubricating oil paste was used as a lubricant during compaction.

Figure 4.1 shows the schematic diagram of the compaction die and punch. Figure 4.2 illustrates the photograph of the compaction processes.
Figure 4.1. Schematic diagram of the Compaction die and punch (Figure not to scale)

Figure 4.2. Photograph of the process of compaction of elemental powder mix
Figure 4.3. Compressibility plots for various alloys
Before compaction of the powder, the following characteristics of the powders were evaluated.

1. Powder particle size i.e., sieve size analysis of the major elemental electrolytic iron powder (Data supplied by Hoganas Ltd, Pune) shown in Table 4.1.

Table 4.1. Sieve size analysis of the elemental Iron powder

<table>
<thead>
<tr>
<th>Sieve Size Analysis- Iron Powder:</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Sieve Size, µm</td>
<td>150</td>
</tr>
<tr>
<td>% Weight Retained</td>
<td>1.42</td>
</tr>
</tbody>
</table>

2. The percentage theoretical densities of the elemental powder mix have been calculated by the equation 4.1.

\[
\rho_{\text{theo}} = \frac{100}{\%A \rho_{\text{theo}}(A) + \%B \rho_{\text{theo}}(B) + \ldots + \%N \rho_{\text{theo}}(N)}
\]

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Table 4.2. Basic characteristics of the mixed elemental powders.

<table>
<thead>
<tr>
<th>Sl.No</th>
<th>Composition</th>
<th>(\rho_{\text{theo}}) (g/cc)</th>
<th>(\rho_{\text{app}}) (g/cc)</th>
<th>(\rho_{\text{tap}}) (g/cc)</th>
<th>Flowability (s/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Fe-0.2%C</td>
<td>7.84</td>
<td>3.15</td>
<td>3.60</td>
<td>0.57</td>
</tr>
<tr>
<td>2</td>
<td>Fe-0.2%C-2%Ni</td>
<td>7.85</td>
<td>3.34</td>
<td>3.53</td>
<td>0.54</td>
</tr>
<tr>
<td>3</td>
<td>Fe-0.2%C-2%Ni-1.5%Mo</td>
<td>7.88</td>
<td>3.25</td>
<td>3.44</td>
<td>0.57</td>
</tr>
<tr>
<td>4</td>
<td>Fe-0.2%C-2%Ni-3%Mo</td>
<td>7.91</td>
<td>3.28</td>
<td>3.45</td>
<td>0.52</td>
</tr>
<tr>
<td>5</td>
<td>Fe-0.2%C-1%Cr</td>
<td>7.82</td>
<td>3.28</td>
<td>3.43</td>
<td>0.57</td>
</tr>
<tr>
<td>6</td>
<td>Fe-0.2%C-2%Cr</td>
<td>7.82</td>
<td>3.25</td>
<td>3.54</td>
<td>0.56</td>
</tr>
<tr>
<td>7</td>
<td>Fe-0.2%C-1%Cr-2%Ni</td>
<td>7.84</td>
<td>3.29</td>
<td>3.53</td>
<td>0.55</td>
</tr>
<tr>
<td>8</td>
<td>Fe-0.2%C-1%Cr-2%Ni-1.5%Mo</td>
<td>7.85</td>
<td>3.28</td>
<td>3.61</td>
<td>0.59</td>
</tr>
</tbody>
</table>
3. The standard Hall flow meter and standard density cup have been used to find flowability, apparent and tap density of the elemental powder mix. Table 4.2 lists the percentage theoretical density, apparent density, tap density and flowability of the elemental powder mix.

4. The required green densities as explained above are achieved by applying the correct pressure during compaction. The correct pressure required for achieving the initial preform green density was known from the compressibility test. The compressibility test results were plotted between applied pressure and percentage theoretical density. Figure 4.3 illustrates the compressibility test graphs of various alloys considered for the present investigations.

4.5. Sintering

Sintering is a thermal process and is done to achieve all possible final strength and hardness needed in the finished P/M product. During sintering bonds are formed between metal powder particles at temperatures below the melting point of the major constituent through atomic transport events [21-24]. There is some shrinkage in dimension during sintering. This generally leads to an increase in density of the consolidated body.

Immediately after the compaction, an indigenously developed ceramic coating was applied on the surface of the compacts twice with a time gap of 24 hours and the compacts were dried for 24 hours. Sintering of the ceramic-coated samples was carried out in a muffle furnace of 3.5 kW capacity at a temperature of 1000 ± 10°C for a period of 2 hours.
4.6. Workability

The relative ease with which a material can be shaped through plastic deformation is called workability [54 & 55]. Workability of a material is usually limited by the onset of fracture. Workability depends not only on the ductility of the material, but also on the stress state of the process. Ease of manufacture is aided when the material has a low flow stress. Grain size and grain structure also influences the workability. Minor variations in composition may also cause large variations in workability, grain size and final mechanical properties.

In order to study the plastic deformation, densification and formability characteristics of the sintered alloys, the following post sintering operations were performed:

1. Cold upsetting of the as sintered preforms
2. Cold repressing of the as sintered preforms
3. Hot upsetting of the sintered preforms

During cold upsetting, cold repressing and hot upsetting, the initial sintered preform density value was fixed in the range 83 ± 2% in order to limit the number of factors governing the densification and deformation of the alloys. Cold upsetting of the sintered preforms was carried out in a hydraulic press of 1000 kN capacity using a set of flat dies and lubricating oil-graphite mixture as a lubricant. Figure 4.4 illustrates the upsetting of the cylindrical preforms. Figure 4.5 shows the photographs of the cold upsetting process performed on the preforms and the different stages of upsetting.
Cold repressing of the preforms has been carried out inside the same die, which was used for compaction of the preforms. The axial applied load was restricted up to the point of material back flow as indicated by a reduction in applied load.
Hot upsetting of the sintered preforms was carried out immediately after the sintering process, using a 2000 kN friction screw press. Figure 4.6 depicts the photographs of the hot upset forged specimens. Upset loads were applied at varying degrees of magnitude at constant ram speed of 0.2 m/s, in order to introduce varying levels of axial deformations on the preforms. Dimensional and density measurements were carried out after each step of cold upset, cold repress and hot upset deformations. Archimede's principle was used to find the actual densities of the deformed preforms after each step of deformation. The axial upsetting was continued up to the point of formation of fine surface cracks on the outer circumference.

4.7. Uncertainty analysis

Uncertainty (error) will creep in any experiment regardless of the care with which experiments are carried out. Hence, in the present work, uncertainty analysis was performed in order to know the uncertainty involved in the present experimental variables such as axial stress, true height strain and percentage theoretical density. The standard deviations obtained in the uncertainty analysis of the experimental results at 99% confidence level are shown table 4.3.
Table 4.3. Results of uncertainty analysis conducted at 250 ± 0.75 MPa, at the average ram speed of 0.75 mm/s.

<table>
<thead>
<tr>
<th>Sl.No</th>
<th>Description</th>
<th>Mean</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Axial Stress (MPa)</td>
<td>418.72</td>
<td>±6.4 MPa</td>
</tr>
<tr>
<td>2</td>
<td>True Height Strain</td>
<td>0.34</td>
<td>±0.01</td>
</tr>
<tr>
<td>3</td>
<td>Percentage Theoretical Density</td>
<td>89.55</td>
<td>±0.17</td>
</tr>
</tbody>
</table>

4.8. Mechanical properties

Immediately after sintering, the cylindrical compacts aspect ratio (A.R) of 1.3 were hot upset forged repeatedly, and drawn in to a square cross section bar of size 10 mm x 10 mm x 110 mm and 12mm x 12mm x 70mm, with near-theoretical densities. Standard tensile (ASTM-E8) and impact (ASTM-E23) specimens were machined off from the hot forged square cross section rods. Hardness measurements on the forged preforms were carried out on a Rockwell hardness tester (ASTM-E18-07). Tensile testing was carried out on a computerized universal testing machine of 1000 kN capacity at a strain rate of 0.5% per min. Standard Charpy impact test was also carried out on the forged alloys. The tensile, impact and hardness values were evaluated for three specimens in each alloy and the mean values of the properties were taken for the present investigations in order to assure the repeatability and authenticity of the results.

4.9. Microstructure, EDX, XRF and Fractography

Microstructural studies on the forged alloys were undertaken using KYOWA, ME-LUX2, microscope fitted with CCD camera and interfaced with a computer and image analyser. EDX was also taken on the alloys in order to estimate the true compositions and also to validate the different phases in the microstructure. XRF data has also been analysed to
find the actual presence of the alloying elements using Innov-X systems (Innovative XRF Technologies). Fractographs of the fractured surfaces of the tensile specimen were obtained using a JEOL-Field Emission Scanning Electron Microscope (TSM-6701F, Japan).