Chapter 3

Materials and Methods

3.1 Introduction

This chapter briefly addresses the experimental techniques employed in developing the in-house set-up, chemical characterization, melting, and pouring of the A308 alloy. It also discusses the physical, microstructural, and mechanical characterization experiments conducted in the current investigation. Characterizations, such as optical microscopy, scanning electron microscopy, energy-dispersive X-ray spectroscopy and X-ray diffraction, are also described. Based on the literature gaps and the steady operational functioning of the mechanical vibration experimental set-up, this work's frequency range has been categorized into low, medium and high-frequency ranges.

The intensity levels of the effect of frequency ranges (mechanical vibration) were increased to assess the impact on the mechanical strength of A308 alloy. Figure 3.1 shows the research work and plans in detail.

3.2 Outline of the investigation

1. A literature review of aluminium alloy casting under mould vibration.
2. To design and development of the experimental vibratory set-up for casting.
3. Material selection for casting.
4. Preparation of castings under stationary and vibratory conditions.
5. Fabrication of test specimens from castings for physical, metallurgical, and mechanical properties.
6. Analyse and correlate the observed results with Archimedes's principle for density, porosity, and hall-petch relation with microstructure using software like Image J.

7. Compare all the results of vibratory casting's physical, metallurgical and mechanical properties with those of stationary castings.

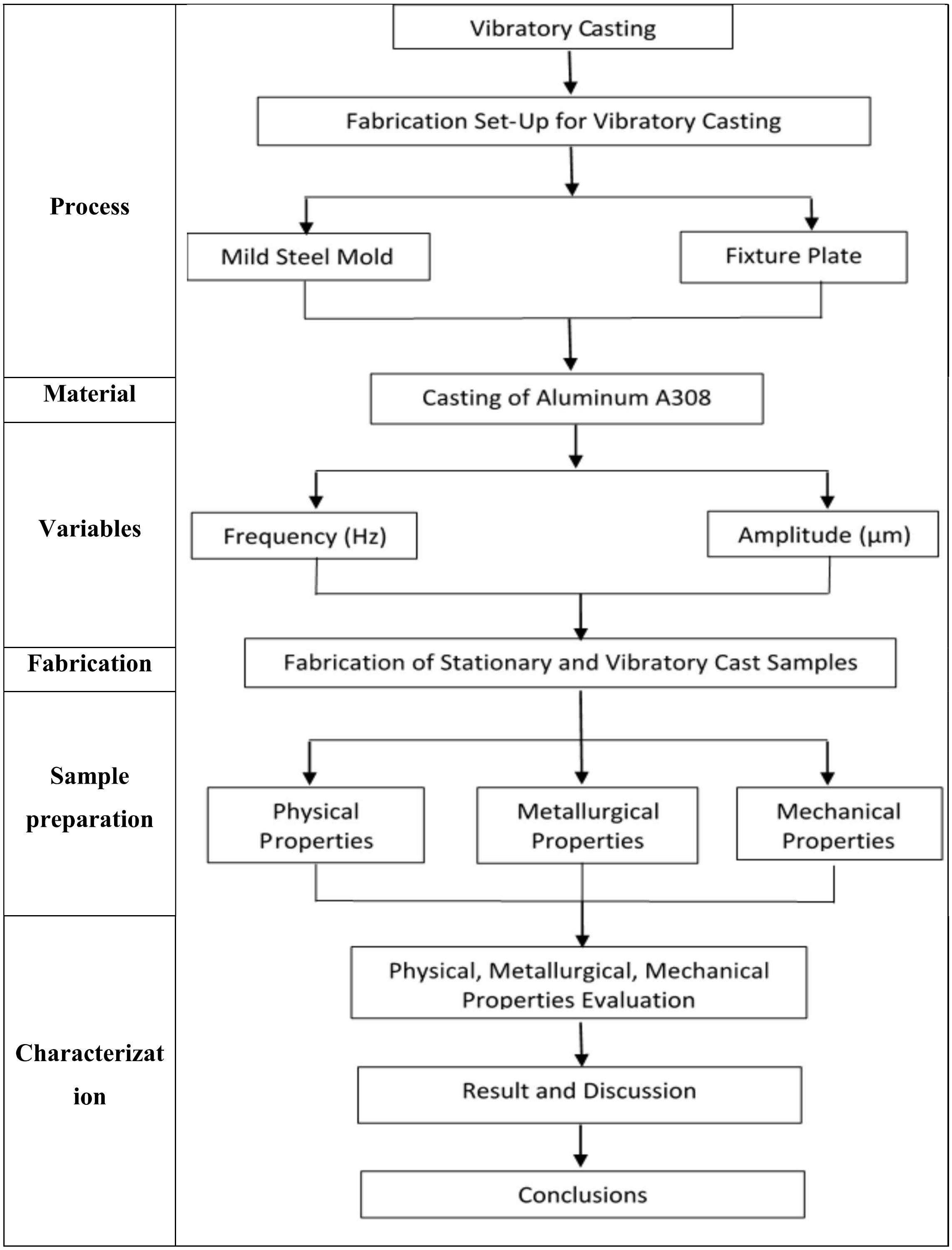

Figure 3.1: Plan of research work

3.2.1 Fabrication of the die

Die design is crucial for producing a high-quality product with a shorter development cycle. The die with optimised design parameters produces complicated castings rapidly with precise dimensional accuracy and a high surface finish, reducing the need for additional machining. Four primary functions of the casting die include (1) holding the molten metal in the required casting shape and (2) ensuring the molten metal can enter the proper holding area. (3) Cool the molten metal until it solidifies; (4) Remove the solidified metal (4) Make provision for the easy removal of the solidified.

Figure 3.2 illustrates the choice of die made after testing mild steel closed die, mild steel open die, and mild steel conical frustum die. Figure 3.2 (a) depicts the mild steel die trial, and it can be seen that the samples had casting flaws including shrinkage and porosity. The open die trial is depicted in Figure 3.2(b), and it can be seen that the samples include casting flaws such as shrinkage and porosity. The conical frustum shape die trial is shown in Figure 3.2(c), and it was found that the cast result is defect-free. As a result, we concluded from these die trials that the conical frustum shape suited best for sound casting, and as a result, we moved forward with the conical frustum shape die.

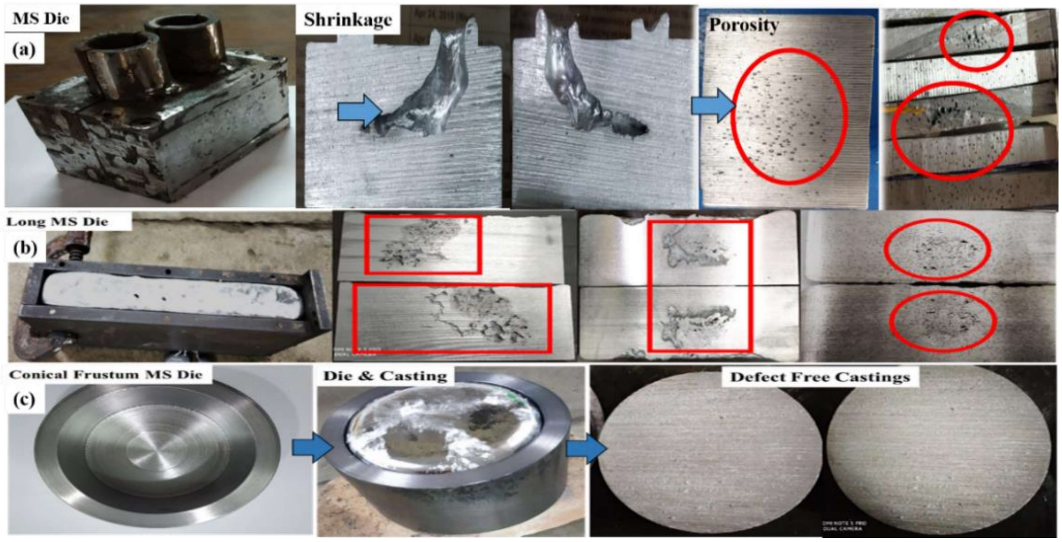

Figure 3.2: Casting trials on mild steel closed die, open die, and conical frustum die

3.2.2 Die and fixture making

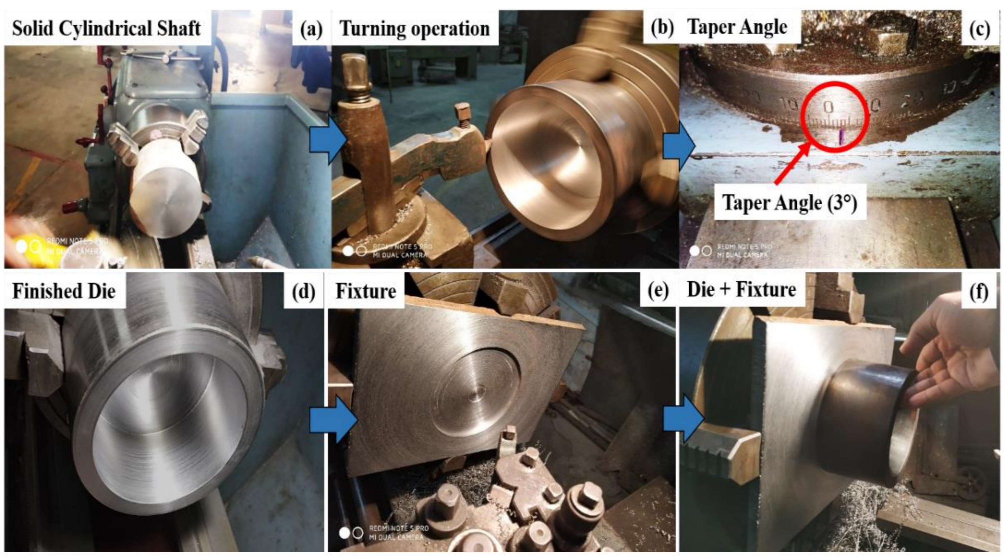

Figure 3.3: Die and fixture making

The fabrication of a mild steel die in the shape of a conical frustum is depicted in Figure 3.3(b) and (d) above. The following steps were taken into consideration when fabricating for the conical frustum shape die, a mild steel solid cylindrical shaft measuring 130x100mm was chosen as shown in Figure 3.3(a). It was then mounted on four jaw chuck lath machines, cleaned up by facing and turning operations using a single point cutting tool, and then finished by internal turning operations. Figure 3.3(d) shows the finished die's dimensions were 115 mm top inner diameter, 110 mm bottom inner diameter, and 70 mm depth. To create a conical frustum shape enabling simple removal of the solidified casting, a draft angle of 3° was applied as depicted in Figure 3.3(d). Figure 3.3(e) shows a fixture plate with the dimensions (300 mm long, 200 mm wide, and 10 mm thick was also created for fixing the die inside a grove of 5 mm, limiting the die's motion under the vibrational effect of frequencies between 2 and 150 Hz, and preventing any potential loss of vibration effect from the vibration generator to the die. Figure 3.2(e) shows a die inserted in a fixture plate.

3.2.3 Fabrication and calibration of horizontal vibration set-up

The fabrication of the setup depicted in Figure 3.4 and 3.5 includes the precise coupling of the exciter to the vibratory table, and two mild steel shafts. Four square hollow rods with dimensions of 410x40x40 mm and 560x40 mm each make up the base plate. These rods were machined using the planer machine. These rods were right-angled welded together to form a rectangular framework with the measurement of 640x410x40 mm. Hollow square rods measuring 40mm x 40mm were utilised on either side to provide the correct height and alignment with the vibrator. Drilled eight holes, each 20mm in diameter, to fix four bearing blocks. 25mm internal bearings are placed in these bearing blocks. On a centre lathe, two mild steel shafts were turned to the measurements of 25 mm in diameter and 410 mm in length. The vibratory table's final dimensions of 380 x 300 x 10 mm were machined. The table was made flat in the fitting shop, and a 'L' plate with a base and vertical plates that measured 100x50x5mm and had a hole of 10mm was bolted to it. The hole was made so that an aluminium bolt could be used to tightly couple the vibratory table to the vibration exciter. The flatness and perpendicularity of the two plates were examined after the two bolts were joined. Two shafts positioned on the bearing above the base plate of the vibratory table.

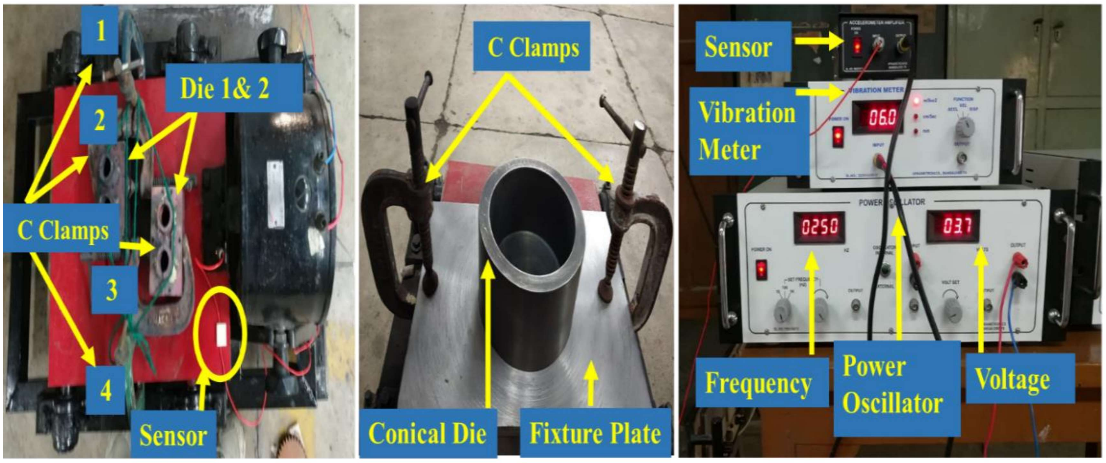

Figure 3.4: Calibration of setup, dry run and amplitude measurement

Figure 3.4 depicts the die and fixtures used for calibration using a dead load and vibration meter. We employed trial-and-error calibrations to achieve constant amplitude for a particular frequency range, and Spranktronics, Bangaluru, the company that made the power amplifier used in the experiment, kept an eye on the proceedings. The calibration procedures are illustrated below and are based on a dry run trial in which the total dead weight was close to (6.224 kg) by maintaining two closed dies (1.136 kg/per die) and four C clamps (0.988 kg/clamp) of equal weight as will be used in real experimental conditions (conical die weight (3.179 kg), fixture weight (1.069 kg), and two C clamp (0.988 kg) die during the casting.

Trial under dry run conditions = 2 dies weight + 4 C clamp (wt.)

= 2*1136 + 4 * 988

= 2.272 + 3.952

= 6.224 Kg

Trial under real conditions = Conical die weight + Fixture weight +2 C clamp (wt.)

= 3.179 + 1.069 + 2*988

= 4.248 + 1.976

= 6.224 Kg

3.2.4 Process design and procedure for frequency and amplitude range selection

Based on data gathered during the calibration of the vibratory set-up by the equipment supplier, Table 3.1 shows the average amplitude for the various frequency ranges (low, medium, and high) (Spranktronics Bengaluru). According to the research gap, with an input voltage of 3.5±0.5, the average amplitude value is 2.5±0.5 mm under a low-frequency range. The average amplitude value recorded was 39±5 µm by increasing the input voltage from 3.5±0.5 to 5.0±0.5, or under medium-range frequency, which is significantly lower than the low-frequency range amplitude value, or 2.5±0.5 mm. The average amplitude value again decreases to 31±5 µm as the input voltage from the power amplifier is increased up to 7.5±0.5 V at 75 Hz frequency. The amplitude did not change much beyond 150 Hz. In light of the observation discussed earlier, three different frequency ranges are selected for the experiment.

Table 3.1: Frequency and amplitude range selection

S/N	Frequency (Hz)	Input voltage (V)	amplitude (mm/µm)	Avg. amplitude (mm/µm)	Frequency range
1	2	3.5±0.5	3	2.5±0.5 mm	Low
2	4		2.5		
3	6		2.5		
4	8		2.5		
5	10		2		
6	20	5.0±0.5	39	39±5 µm	Medium
7	30		41		
8	40		38		
9	50		39		
10	75	7.5±0.5	36	31±5 µm	High
11	100		31		
12	125		29		
13	150		27		

The horizontal vibration casting set-up was used to cast the A308 alloy and die was vibrated horizontally during casting using a mechanical vibration system that included a power amplifier, vibration generator, base plate, angular plate, vibratory plate, and fixture plate as shown in Figure 3.5. Inside the rectangular mild steel fixture plate (300 mm length, 200 mm width, 10 mm thickness), a conical frustum shape metallic die (115 mm top inner diameter, 110 mm bottom inner diameter, 70 mm depth) was fixed. To keep the die tightly inside the plate, a 5 mm deep groove was carved across the 135 mm diameter of the fixture plate. The fixture plate was then fastened to the vibratory table with a set of C-clamps, preventing relative motion between the table and the die.

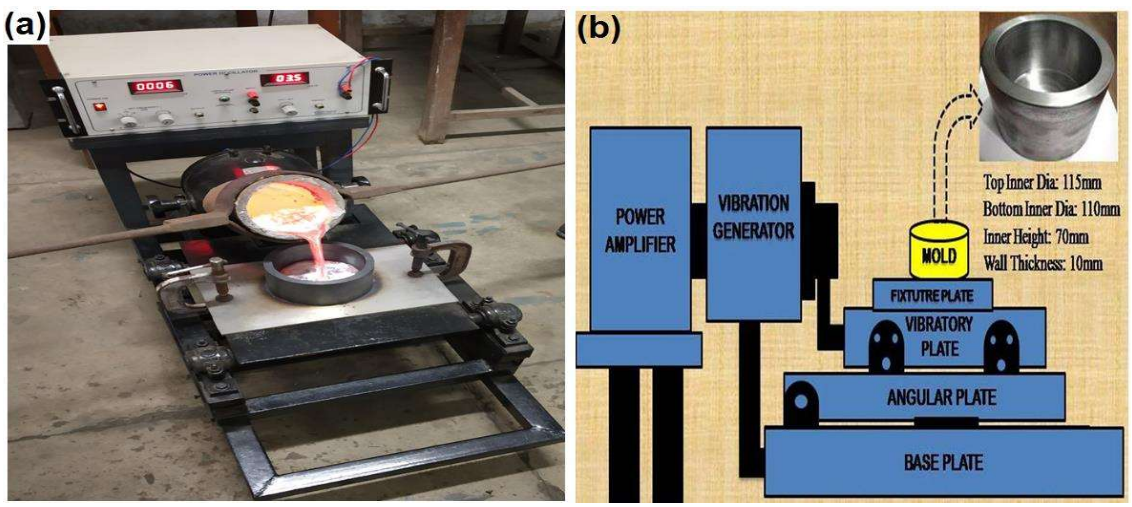

Figure 3.5: Casting set-up (a) horizontal mechanical vibration M/C and (b) Schematic diagram

3.2.5 Selection of material

In this study, A308 (LM21) alloy was chosen as the experimental material because of its availability and excellent castability. This alloy was procured from a local supplier in Varanasi (India). A308 alloy has not yet been commercialized because of the absence of improvements in mechanical strength and physical characteristics. The A308 alloy can be used as a substitute for the A319 alloy because of their similar behaviour. Commercial aluminium alloys, such as A356 or A319 alloy, is used in aircraft and automotive components. The chemical composition of the A308 alloy was determined using an optical emission spectrometer (Foundry Master), as indicated in Table.1.

Table 3.2: Chemical composition of A308 Alloy by wt.%

Elements	**Si**	**Cu**	**Mn**	**Fe**	**Mg**	**Ti**	**Zn**	**Al**
As per BS 1490:1988 Std.	5.0-7.0	3.0-5.0	0.2-0.6	1.0	0.1 - 0.3	0.2	2.0	Bal.
Wt. %	5.45	3.52	0.60	0.72	0.04	0.01	0.04	Bal.

3.2.6 Pouring of casting of samples poured under stationary and vibratory condtions.

The melting of A308 alloy (2 kg) was performed in an electric resistance furnace at 750°C. Degassing was done using hexachloroethane (C_2Cl_6) as the flux of 0.6wt.% (12gm) of molten metal before pouring it into the metallic mould. It primarily prevents the trapping of gases, the development of oxides, and the dissolving of hydrogen gas. Finally, the molten metal was poured into a preheated 200°C metallic mould at a pouring temperature of 710°±10°C as shown in Figure 3.6. Castings prepared under horizontal mechanical vibration at low (0, 2, 4, 6, 8, and 10 Hz), medium (0, 20, 30, 40, and 50 Hz), and high frequencies (0, 75, 100, 125, and 150 Hz) constant amplitude 2.5±0.5mm, 39±5µm, and, 31±5µm respectively are shown in Figure 3.7 to 3.9. The time duration between the pouring temperature (710°C) and freezing temperature (520°C) of each casting was measured while mechanical vibration was applied. The cooling rate (degrees celsius per second) was computed by multiplying the temperature difference (degrees Celsius) by the solidification time (Sec.). The final cast components were obtained after successful pouring, as shown in Figs. 3 and 4.

Figure 3.6: Melting, die preheating, die & fixture assembly and pouring

Figure 3.7: Cast components under a low-frequency (0-10) Hz range

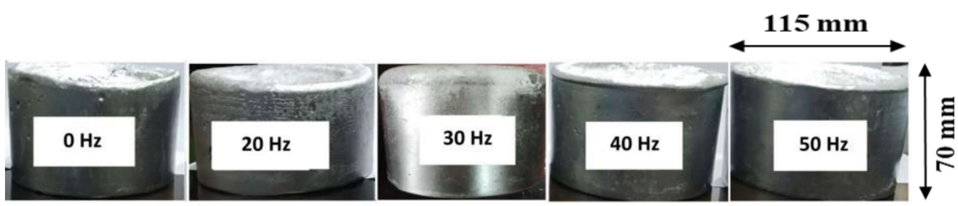

Figure 3.8: Cast components under a medium-frequency (0-50) Hz range

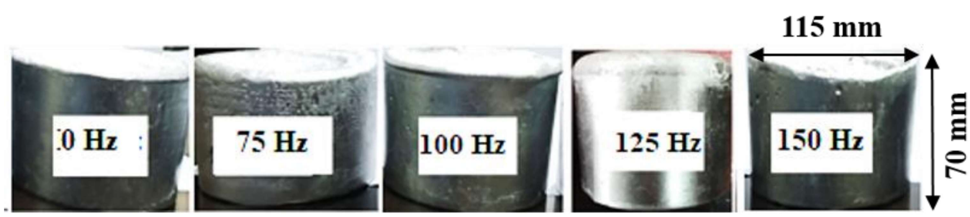

Figure 3.9: Cast components under a high-frequency (0-150) Hz range

3.3 Characterizations

For various characterizations, specimens were extracted from as-cast and vibrated conditions. Figure 3.10 depicts the precise locations of sample extraction for physical, metallurgical and mechanical tests samples. While optical and scanning electron microscopy were used to study the microstructural details, optical emission spectroscopy and X-ray diffraction were used to analyse the composition. Density, microhardness, and tensile testing, for the specimens of stationary and vibratory castings, were among the tests carried out to evaluate the performance of the castings.

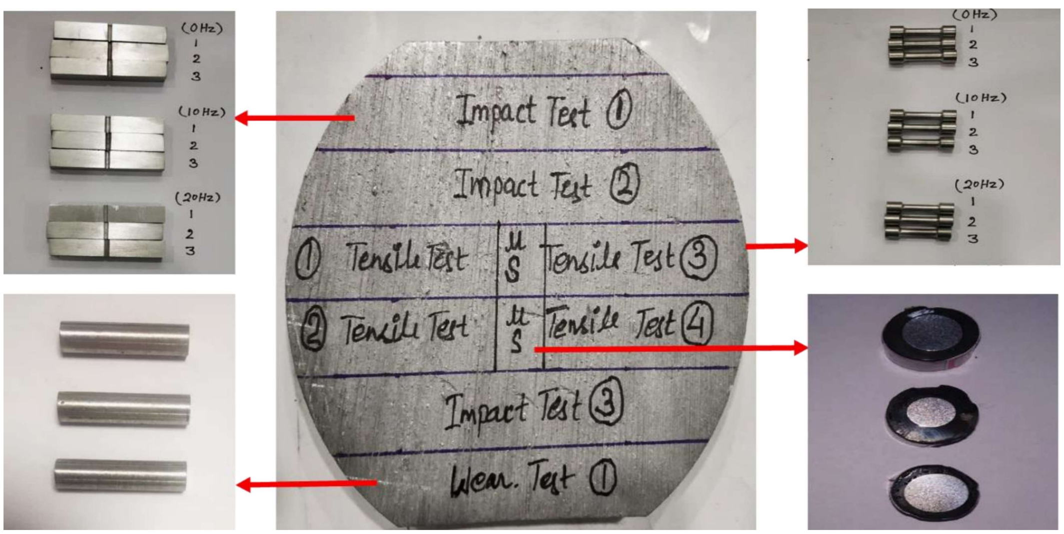

Figure 3.10: Sample extraction location for physical, metallurgical and mechanical test samples

3.3.1 Machines used for characterization

1. Optical Microscope: (Leitz Metallux-3)
2. Scanning Electron microscope: (Serial no.-EVO18- 20-45, ZEISS EVO 18 Research, Germany)
3. Energy Dispersive X-ray Spectroscopy (EDS-51N1000, Oxford Instruments, UK)

4. X-Ray Diffraction:(Model- MINIFLEX 600 DETEXULTRA, Japan)
5. Microhardness Tester: (Model- LECO 600 DETEX ULTRA, Japan)
6. Tensile Testing Machine: (A computerized 100 kN screw-driven Instron™ Universal Testing Machine, model 5848)

3.3.2 Density and % porosity measurements

A digital density balance (CAH-503, Contech Instruments, India) with a measurement accuracy of 0.0001g/cc was used to quantify the experimental density of cast specimens, as illustrated in Figure 3.11.

Figure 3.11: Density measuring instrument

The experimental density of cast specimens was calculated using Archimedes' method. The experimental density of each sample was determined after measuring the weight of each specimen independently in air and water as the given equations 1 & 2:

$$\rho_{exp} = \frac{W_a}{W_a - W_L}\rho_L \tag{3.1}$$

Where, ρ_{exp} is the experimental density, W_a is the weight in the air, $W_L$is the weight in water and ρ_L is th**e** density of distilled water [28].

Here, the rule of mixture for alloys [29] was applied to calculate the theoretical density of different specimens of cast A308 alloy as per equation (2) given below:

$$\rho_{th} = \frac{1}{\sum \frac{w_i}{\rho_i}} \times 100 \quad (3.2)$$

Where W_i is the weight fraction of i^{th}(Si, Cu, Mn, Fe, Mg, Ti, Zn, V, and Al) elements mentioned in chemical composition analysis presented in Table 1 and ρ_i denoted as the theoretical density of i^{th} elements. Using the following formula (3), the value of % porosity was computed using theoretical density (ρ_{th}) and experimental density (ρ_{exp}):

$$\text{(\%) Porosity} = \frac{\rho_{exp} - \rho_{th}}{\rho_{th}} \quad (3.3)$$

3.3.3 Optical microscope

Metallographic specimens were extracted from the central region of the castings are shown in Figure 3.12 and prepared according to the ASTM E3 standard. Specimens were polished to a mirror finish using emery paper and a microfiber cloth. Before metallographic testing using an optical microscope, the samples were etched using Keller's reagent (5 mL HNO_3, 3 mL HCl, 1 mL HF, and 190 mL H2O). ImageJ analysis software was used to measure and analyse metallurgical features, such as primary-Al grain size, eutectic silicon size, form factor, aspect ratio, percentage roundness of eutectic Si, and SDAS, using the linear intercept approach.

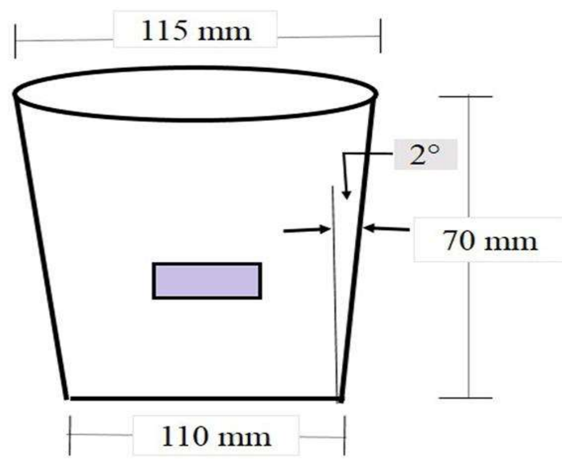

Figure 3.12: Sample sectioning for microstructural examination

3.3.4 Scanning electron microscope

Using a scanning electron microscope (EVOSEM18, ZEISS, Germany) equipped with energy-dispersive X-ray spectroscopy (EDS-51N1000, Oxford Instruments, UK), the morphologies of precipitates, such as Al_2Cu, Fe-intermetallics, and eutectic Si phases, were examined.

3.3.5 X-ray diffractometer

Using an X-ray diffractometer (Rigaku Miniflex 600 DTex Ultra) with a Ni filter, an X-ray diffraction investigation was performed to identify constituents and intermetallic phases contained in the A308 alloy.

3.3.6 Tensile and microhardness test

Mechanical properties of the cast samples, such as tensile strength and Microhardness, were examined according to standard dimensions. Tensile specimens were prepared in line with ASTM standard E8 (4.5 mm gauge diameter and 15.5 mm gauge length) for all castings to observe tensile strength. The tensile test was carried out on Instron-5848 tensile testing equipment with a crosshead speed of 1.0 mm/min. Microhardness tests were performed on metallographic specimens using Vickers hardness test equipment and a square pyramid indenter at a force of 10 N and a dwell length of 10 seconds. For each sample, the microhardness tests were repeated five times, with the average values provided.

3.3.6.1 Preparation of tensile test specimen

As indicated in Figure 3.13, tension test specimens were made from casting workpieces according to ASTM requirements. A lathe machine was used to machine the circular cross-section test pieces. The ends were shaped to fit the tensile testing machine's grasping gear. The end was left long enough to allow for appropriate gripping. The length of the test specimen was sufficient to ensure that necking did not occur towards the ends. All surface defects were removed by machining the test specimen, and the original dimension was retrieved using emery paper.

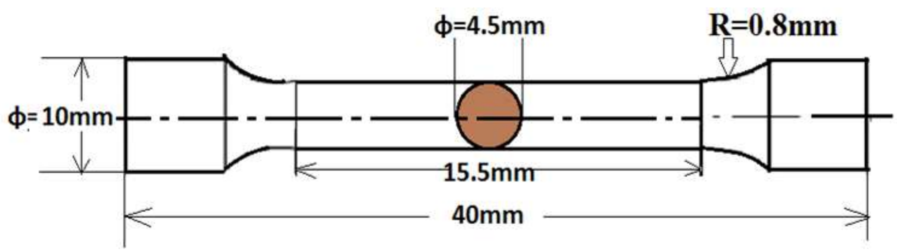

Figure 3.13: Tensile test specimens

3.3.6.2 Preparation of microhardness test specimen

Microhardness tests were carried out to measure each sample’s hardness as shown in Figure 3.14 and value against deformation resistance. On a macroscopic or microscopic scale, the hardness tests were carried out at room temperature for each sample. The hardness values of each sample were correlated to their tensile strength, which is linearly proportional to each other. This fundamental relationship allows for cost-effective and non-destructive testing for bulk metal using portable equipment like Vickers microhardness testers. The specimen is crushed with the indenter with a dwell period of

10 seconds during the 10 N test force. The indenter is released, producing a square-shaped impression within sample surfaces. The indenter size can be measured optically simultaneously as the experiment looks at the square impression with two diagonals. The Vickers hardness number is calculated by dividing the test force by the indenter surface area. The Vickers hardness values were calculated using the formula's average of the diagonals in the experiment of hardness measurement. The loading and unloading of the load are done automatically. The Vickers microhardness HV is calculated using the given formula as expressed below,

$$HV = 1.854F / d^2$$

Where F is the load applied in kgf, d is the mean of the two diagonals, d_1 and d_2 in mm HV is Vickers hardness, and Dwell time = 10 seconds.

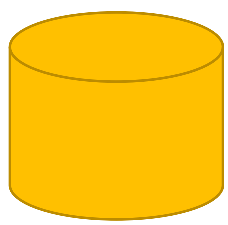

Figure 3.14: Microhardness specimen

3.3.7 Characterization & correlation of the observed results using microstructure

The microstructural analysis was carried out using the ImageJ software tool, which was included with the optical microscope used to capture and analyse the pictures. At 100 magnifications, a snapshot of the microstructure of a test specimen was taken from several locations. With the help of Image analysis software (ImageJ software tool), the ASTM micro-grain size was measured from the obtained image using line intersect methods.

3.3.8 Fractography of tensile test samples

Scanning Electron Microscopy (SEM) in combination with EDX (ZEISS Model-EVO 18) analysis has been used to investigate the fracture behaviour of test samples.

3.4 Summary

The experimental design and characterization methods are covered in detail in this chapter. The outline of the experimental investigation, die fabrication, a brief discussion of die and fixture design, calibration of the setup, frequency and amplitude range selection, equipment set-up design, melting, pouring, and equipment schematics, selection of material, casting of samples under stationary and different vibratory ranges, machines used for characterization, characterization, density and % porosity measurements, tensile and microhardness test, characterization and correlation of the observed results using microstructure and ImageJ software tool, fractography of tensile test samples have all been included. To determine the quality of casting based on physical, metallurgical, and mechanical characteristics concerning variation in the frequency and amplitude of horizontal mechanical vibration casting technique, the aforementioned experimental and investigative process is used. Future chapters will contain this general division of low, medium, and high for the frequency and amplitudes.