3 MATERIALS AND METHODS

3.1 Material selection

Hardened AISI D2 tool steel as a work material was used in the present experimental work to conduct surface grinding experiments. The reason for selecting AISI D2 tool steel is that it has a wide application in the industries, i.e., mold and die, aerospace, automotive, medical appliances, heavy engineering and tools manufacturing industries [203]. It comes under difficult-to-cut material used for manufacturing: pneumatic tools, press tools, chisels, bending and forming dies, deep drawing cold extension dies, gauges, collets and punches, etc., owing to its greater wear-resistant quality, non-deforming characteristics and high hardness after hardening methodology [204]. The chemical composition of the as-received work material is obtained by energy-dispersive X-ray spectroscopy (EDS), as presented in Table 3.1, and Table 3.2 provides detail on the mechanical properties of the AISI D2 tool steel used in the current experiments.

Table 3.1: The elemental composition of AISI D2 tool steel (wt.%), the remaining is Fe.

Element	C	Cr	Ni	Si	Mn	Mo	V	S	P	Cu
wt.%	1.422	11.344	0.201	0.447	0.366	1.097	0.80	0.003	0.015	0.029

Table 3.2: Mechanical properties of AISI D2 tool steel [205], [206].

Properties	**Value**	**Properties**	**Value**
0.2% offset yield strength (MPa)	1532	Poisson’s ratio	0.30
Hardness (HRC)	58±2	Thermal conductivity(W/mK)	21
Young’s modulus (GPa)	180	Specific heat (J/kg-K)	485
Density (kg/m^3)	7750	Melting point (°C)	1460

The grinding experiment employed the cuboid shape of work material 80 × 15 × 8 mm^3 in size. Each work material was subjected to heat treatment at an outdoor heat treatment facility (Metals Heat Treatment, India) to achieve a 58±2 HRC hardness for industrial applications. The work materials were initially heated gradually to a temperature of 650 °C and then kept there until they were all equally heated. The work materials were heated further until they reached 1050 °C and were kept there for 90 minutes inside a vacuum furnace (SECO-WARWICK, India). After that, the work materials were quenched in oil. The power source for this furnace was 230 V, 50 Hz, and the quenching pressure was kept between 5-8 bar. Finally, work materials were first tempered at 400 °C for 120 minutes and then tempered again at 450 °C for 120 minutes to improve toughness and relieve internal stresses caused by the quenching process. Fig. 3.1 shows as received work material and microstructure of that material.

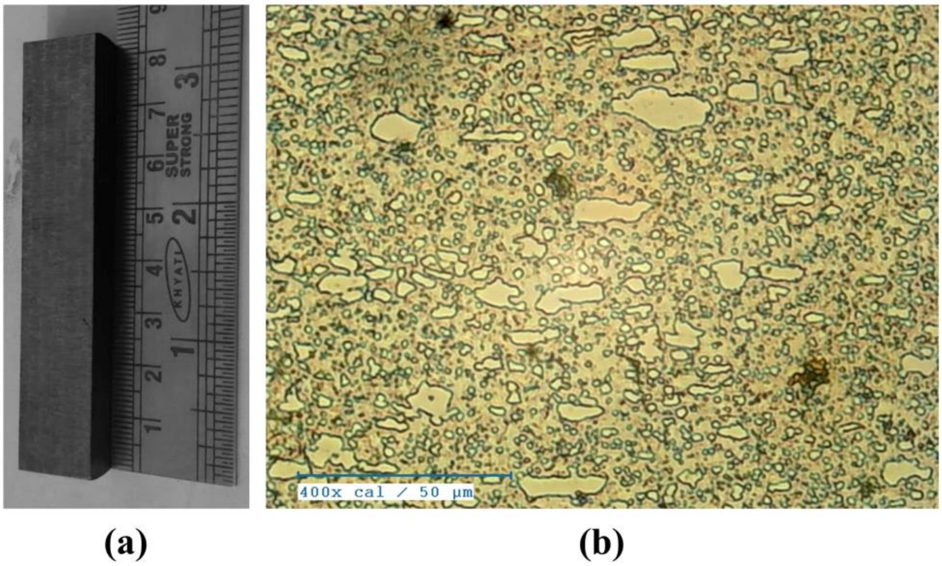


Fig. 3.1: (a) As-received work material; (b) Microstructure of AISI D2 tool steel.

3.2 Grinding wheel

The grinding experiments were conducted on the surface grinding machine (455 H, Hindustan Machine Tool Ltd., India) using conventional aluminum oxide (Al_2O_3) grinding wheel (AA-60-K-5-V) of size 250 × 20 × 76.2 mm^3. This wheel has medium-grade, medium-abrasive grain size and vitrified bonds (refer to Fig. 3.2). Based on the dressing of the wheel and economic concerns, a conventional Al_2O_3 grinding wheel is suitable for grinding AISI D2 tool steel. Aluminum oxide, often known as corundum, is the most well-known and widely used abrasive and is frequently employed with ferrous materials, especially steels. The abrasive grits are the main component of a grinding wheel that affects how well it grinds. Various substances (chromium oxide, titanium oxide, etc.) may be added to enhance hardness, toughness, etc. All the grinding tests were carried out under the up-grinding mode. Many trial tests were done on the work material to determine the optimal process parameters. Therefore, to keep the uniformity in the wheels' topography, after each trial, the grinding wheel was dressed by a single point diamond dresser of size one carat (1 carat = 0.2 g) to a consistent depth of 10 µm up to 50 µm.

Fig. 3.2: Aluminum oxide grinding wheel.

3.3 Experimental setup and conditions

This section is categorized into three subsections as mentioned below:

3.3.1 Cryogenic grinding system

An indigenous cryogenic cooling setup was developed to study the effect of grinding parameters on the workpiece, i.e., tool steel (AISI D2), under a cryogenic environment. This setup consists of two major components: (a) nitrogen gas cylinder and (b) TA-55 liquid nitrogen container (Dewar), as shown in Fig. 3.3.

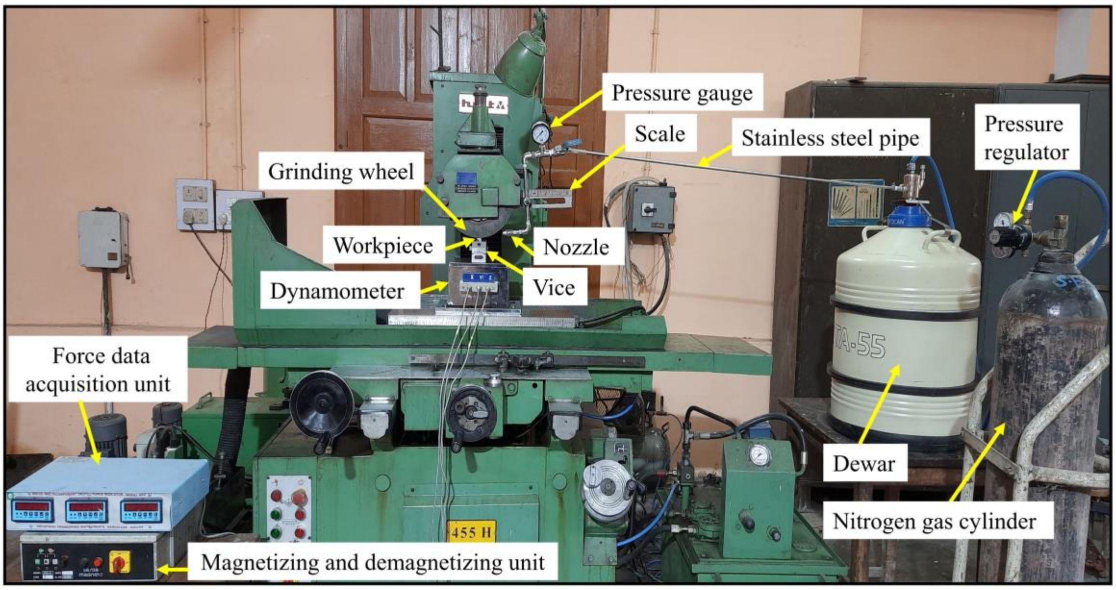


Fig. 3.3: Cryogenic grinding experimental setup used for grinding of AISI D2 tool steel.

A liquid nitrogen container, or Dewar, has a storage capacity of 51.5 litres of liquid nitrogen. It is composed of aluminum, and the walls are completely sealed because the LN_2 inside the Dewar cannot be impacted by ambient temperature. Dewar is closed with a stainless steel cryogenic container cap at the top (refer to Fig. 3.3). Fig. 3.4 shows the cryogenic container cap. An inlet and outlet pipes, which are made of stainless steel, are inserted into the Dewar through the top opening. The main work of these pipes is to deliver the nitrogen gas into the Dewar and to supply the liquid nitrogen to the grinding zone area. A nitrogen gas cylinder is placed before the Dewar, which is used to deliver the compressed nitrogen gas to generate the required pressure in Dewar for supplying the liquid nitrogen to the grinding region through the nozzle via a stainless pipe.

The cryogenic system uses a flat spray nozzle (Spraytech Systems India Pvt. Ltd., India) with a 3 mm exit diameter to inject LN_2 cryogen with a flow rate of 0.2 l/min into the grinding zone area between abrasive wheel-workpiece material. A 12° nozzle angle and a 50 mm stand-off distance were maintained throughout the experiments. The descriptions of the grinding parameters are summarized under cryogenic cooling in Table 3.3.

Fig. 3.4: Cryogenic container cap.

Table 3.3: Grinding parameters adopted during cryogenic grinding experiments.

Parameters	Details
Grinding machine	Hydraulic surface grinder (455 H, HMT)
Grinding mode	Up grinding
Grinding wheel	Aluminum oxide (Al_2O_3)
Wheel specifications and dimensions	AA-60-K-5-V and 250 (outer dia.) × 20 (width) × 76.2 (bore dia.) mm^3
Workpiece material and dimensions	AISI D2 tool steel and 80 (length) × 15 (width) × 8 (thickness) mm^3
Downfeed (a_p)	10–40 µm in step of 10 µm
Table feed rate (V_w)	6-12 m/min in step of 3 m/min
Wheel speed (V_s)	39.42 m/s
Grinding environments	Dry, wet, and cryogenic (LN_2)
Nozzle angle	12°
LN_2 nozzle orifice diameter	3 mm
Stand-off distance	50 mm
LN_2 coolant pressure (P_{LN_2})	2-4 bar in step of 1 bar
LN_2 flow rate (Q_{LN_2})	0.2 l/min
Dresser	Single point diamond dresser
Dressing depth	10 µm in single pass
Dressing environment	Dry

The parameters were set to allow the LN_2 into the interface region between the abrasive wheel and workpiece while minimising the effect of the stiff air layer (hydrodynamic boundary layer) around the abrasive wheel during the grinding operation. The cryogenic coolant, i.e., LN_2, spray pressure supplied to the interface area was fixed at 2, 3, and 4 bars. A pressure gauge controlled this pressure, which produced better cooling and lubrication results. Based on the preliminary tests, process parameter (i.e., wheel speed, downfeed, table feed rate, etc.) conditions are chosen for grinding experiments under all environments. The experiment outputs were noted after three passes with five repetitions for all the trials. For the analysis, an average value of each trial was taken.

3.3.2 Cryo-MQL grinding system

An in-laboratory fabricated economic Cryo-MQL setup was used in the present study to enhance the cooling and lubrication effects during the grinding operation. The Cryo-MQL technique combines cryogenic cooling and MQL. The Cryo-MQL system includes a dewar, nitrogen gas cylinder, air compressor, pressure gauge, flow control device, air control valve, MQL air atomizing spray nozzle, and LN_2 flat spray nozzle. This system was integrated with the 455 H HMT surface grinding machine, as shown in Fig. 3.5. Fig. 3.5 shows the schematic diagram and real image of the indigenous Cryo-MQL experimental setup for the grinding process.

An MQL atomizing nozzle and LN_2 nozzle (Spraytech Systems India Pvt. Ltd., India) are made of stainless steel with an orifice diameter of 1 mm and 3 mm at a 40 mm and 50 mm stand-off distance, respectively. MQL aerosol and LN_2 spray are simultaneously directed to the grinding zone using the hybrid Cryo-MQL technology. The air atomizing spray nozzle has two inlets: (i) to deliver the prepared lubricant and (ii) to supply the compressed air.

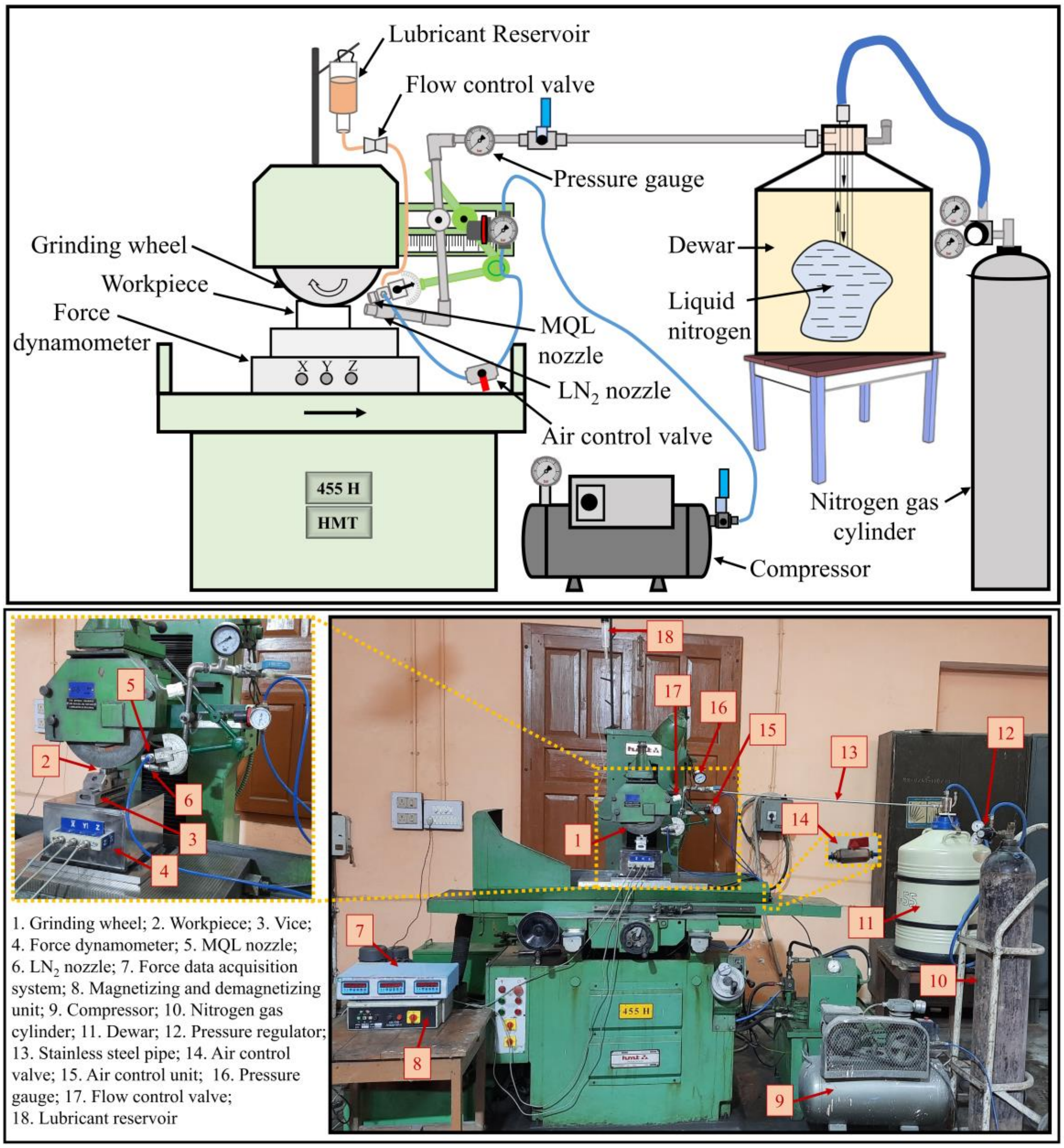


Fig. 3.5: Schematic diagram and real image of Cryo-MQL experimental setup used for grinding of AISI D2 tool steel.

In the MQL method, lubricant is delivered from the reservoir to the grinding zone by gravitational method, and compressed air with high pressure is used as a carrier for lubricant. The flow rates of lubricant and compressed air are controlled separately by a flow control valve and an air control valve, respectively. Firstly, lubricant, and compressed air are mixed in the mixing chamber of the nozzle and, after that, delivered to the grinding zone. An air compressor is a major part of MQL systems. Generally, the air compressor in

MQL continuously supplies pressurized air to the lubricating system via an air hose pipe. The lubricant is atomized using this pressurized air and then sprayed onto the cutting surfaces in a very fine mist. The detailed function of the cryogenic grinding system has been discussed in section 3.3.1. Table 3.4 lists grinding parameters for Cryo-MQL environments.

Table 3.4: Grinding parameters adopted during Cryo-MQL grinding experiments.

Parameters	**Details**
Grinding machine	455H HMT Surface grinder
Grinding mode	Surface grinding (Up grinding)
Wheel speed (V_s)	39.42 m/s
Table feed rate (V_w)	9 m/min
Downfeed (a_p)	40 µm
Grinding environments	MQL and Cryo-MQL
MQL mist flow rate of vegetable oil (Q_o)	200 ml/h
MQL mist flow rate of vegetable oil-based deionized water emulsion and nanofluids (Q_e)	150 ml/h
Air pressure (P)	4 bar
Stand-off distance	40 mm for MQL nozzle and 50 mm for LN_2 nozzle
Spray angle	10-20°
MQL lubricant	Soybean oil (SO), soybean oil-based deionized water emulsion (SO+DW), 0.5 wt.% Al_2O_3 NFs, and 1 wt.% Al_2O_3 NFs
Liquid nitrogen	P_{LN_2} = 2 bar, Q_{LN_2} = 0.2 l/min

3.3.3 Magnetic Barkhausen noise analyzer

Magnetic Barkhausen noise analyzer is a non-destructive technique (NDT) device. This analyzer consists of a magnetic probe, controller, and computer device. The MBN measurements were carried out on ground samples using a commercially available magnetic Barkhausen noise analyzer supplied by Technofour Magstar, India (refer to Fig.

3.6). Table 3.5 represents the details of magnetic BN and HL input conditions for the characterization of the ground sample (AISI D2 tool steel).

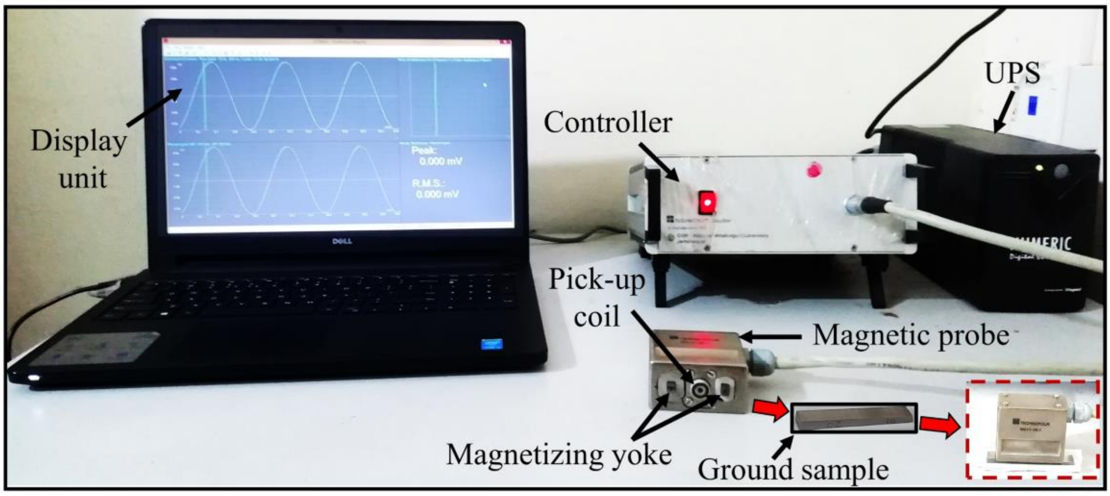


Fig. 3.6: MBN measurements by magnetic Barkhausen noise analyzer.

Table 3.5: BN and HL parameters used in the magnetic characterization for ground sample.

	Parameters	**Details**
BN	Filter	100-300 kHz
	Gain	20 dB
	No. of cycle	3
	Magnetizing frequency	25 Hz
	Magnetic field intensity	200, 300, 400, and 500 Oe
	Outcomes	RMS, peak, and MBN envelope
HL	Waveform	Sinusoidal
	Magnetizing frequency	0.05, 0.1, 0.15, and 0.2 Hz
	Magnetic field intensity	200, 300, 400, and 500 Oe
	Outcomes	HL envelope characteristics: Average permeability, coercivity

The MBN technique is used to analyze the surface integrity of the ground samples. A cuboid shape type magnetic probe with two magnetizing yokes and a pick-up coil at the centre was used to create a magnetic field into the ground samples and receive the BN signal, respectively. The ground surface of the sample was cleaned with acetone before the measurement of Barkhausen noise (BN) and hysteresis loop (HL) responses. For MBN measurement, the location was selected at the central area of the samples. The RMS, peak, MBN envelope, average permeability, coercivity, and HL profile were chosen for analysis in the present study. The average value of five observations for each sample (analyzed by Magstar software) was taken for analysis.

3.4 Grinding environments

Grinding operations investigations were carried out on the AISI D2 tool steel with alumina grinding wheels at various values of table feed rate and downfeed in dry, wet, and cryogenic cooling, MQL and Cryo-MQL conditions in a hydraulic surface grinder.

3.4.1 Dry grinding

In dry grinding, the grinding operation was performed under atmospheric conditions without a coolant. It is the simplest method but can generate significant heat, leading to faster tool wear and the potential for thermal damage to the workpiece.

3.4.2 Wet grinding

In the wet grinding, 20% water-based coolant oil from the coolant tank was pumped and supplied through the reinforced hose with a conical nozzle of 10 mm exit diameter at a 1.5 l/min flow rate. It has a composition of 80% water and 20% soluble oil. Therefore, the coolant spray helps reduce friction and heat at the grinding zone, improving tool life and surface finish.

3.4.3 Cryogenic grinding

When using cryogenic cooling, a nozzle of 3 mm diameter was used to supply the LN_2 coolant through the stainless pipe at the interface of the grinding wheel and the work material in the range of 2-4 bar pressures with a standard nozzle angle of 12° and a stand-off distance of 50 mm. The flow rate of LN_2 coolant was 0.2 l/min. The external supply of the LN_2 coolant is the most favourable method for reducing cutting temperature.

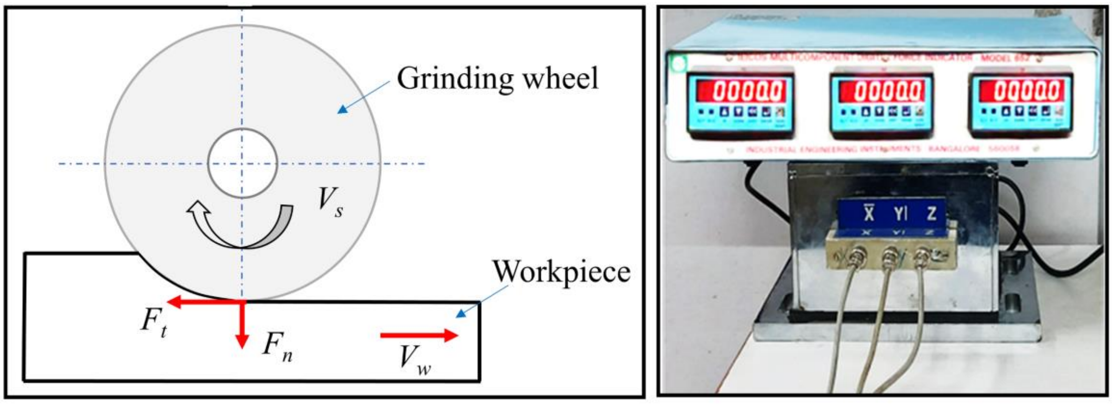
3.4.4 MQL and Cryo-MQL grinding

MQL grinding involves the delivery of lubricants (SO, SO+DW, Al_2O_3 (0.5 wt.%), and Al_2O_3 (1 wt.%)) directly at the interface of the grinding wheel and work material in the range of 10-20° angles with a sufficient pressure of 4 bar and a stand-off distance of 40 mm. Similarly, combining cryogenic and MQL, i.e., hybrid Cryo-MQL grinding, supplies the LN_2 and lubricants at the grinding zone via a separate nozzle following cryogenic and MQL grinding conditions as discussed above.

3.5 Observations regarding the grindability and surface integrity indices

3.5.1 Grinding forces and specific grinding energy

Grinding force is a vital response parameter to investigate any engineering material's grinding behaviour. The grinding action generates forces between the grinding wheel and the workpiece. Grinding forces values provide primary insights into the efficacy of grinding operations. This includes an examination of wheel loading behaviour, as well as the ability to forecast grinding zone temperature, coefficient of friction, specific grinding energy, and surface roughness. Grinding forces are separated into two components: (i) tangential component (F_t) and (ii) normal component (F_n), as seen in Fig. 3.7 (a). Both components are measured by a three-dimensional force 610C IEICOS dynamometer (Industrial Engineering Instruments, India) with an accuracy of ±1%, sampling rate of 1000 Hz, a response time of 1 millisecond and a frequency filter range of 0–45 kHz. The dynamometer consists of X, Y, and Z component force sensors, as shown in Fig. 3.7 (b). This dynamometer has a range of force up to 500 kg force in the XYZ direction. X and Z components represent tangential force and normal force, respectively. Filtration of grinding force signals was done with dynamic grinding force software (3-channel model 652 system). This software plotted a graph between force components and time. This dynamometer is kept over the magnetic chuck of the grinding machine. Magnetic chuck fixes the dynamometer. After that, to measure the grinding force, AISI D2 tool steel work material was clamped over the dynamometer. On the other hand, the accurate estimation of the coefficient of friction in the grinding process is challenging due to its dynamic and complex nature. Thus, the apparent coefficient of friction has been determined by computing the ratio of the tangential force to the normal force.



(a) (b)

Fig. 3.7: (a) Schematic diagram of grinding forces; (b) Three-component dynamometer.

Specific grinding energy is the most important response variable throughout the material removal process, especially when grinding difficult-to-machine materials. “Amount of energy required for removing a unit volume of material” is called specific grinding energy (U_g). During the grinding process, specific grinding energy is significantly related to the grinding wheel service life and the surface quality of the workpiece. This energy also signifies the lubricating action occurring in the grinding zone region. A decrease in specific grinding energy during the process indicates more efficient workpiece machining and is environment-friendly. The dynamometer is used to measure the tangential grinding force, and that tangential force was put in the following equation for calculating the U_g. The following formula for specific grinding energy is given below [207]:

$$U_g = \frac{F_t \cdot V_s}{V_w \cdot a_p \cdot b} \tag{3.1}$$

where U_g: specific grinding energy (J/mm^3), F_t: tangential grinding force (N), V_s: grinding wheel speed (m/s), V_w: table feed rate (mm/s), a_p: downfeed (mm), and b: grinding wheel width (mm).

3.5.2 Grinding temperature

The grinding temperature has a significant impact on the grinding process. It changes the physical properties of the workpiece and the grinding wheel, affecting the material removal rate, surface quality, and overall efficiency of the grinding operation. During the grinding process, friction between the abrasive wheel and workpiece produces heat that leads to wear and tear of the abrasive wheel and deteriorates the workpiece's surface quality in terms of poor surface finish. Hence, analysing the grinding temperature produced while grinding AISI D2 tool steel is essential. The grinding temperature was taken by a non-contact thermal imaging camera (FLIR E75). The infrared thermal imager has 320 × 240 pixels infrared resolution and 30 Hz frame rate. It is set to a measurement range of -20 to 650 ºC, an emissivity of 0.95 with thermal accuracy of ±2%. After performing several trial experiments, the thermal imaging camera was fixed at an optimal distance of 550 mm from the grinding zone using a tripod for proper focus on the grinding zone, as shown in Fig. 3.8.

Fig. 3.8: Temperature measurement arrangement during grinding.

3.5.3 Surface roughness

Surface roughness provides information about the surface characteristics of the processed/machined component. It is a section of surface texture used to determine the peaks and valleys characteristic patterns on the machined workpiece surface. These characteristics reflect the surface quality of the workpiece after the machining operation.

The surface roughness in the perpendicular direction of the grinding path was measured using a high-precision surface profilometer (Mitutoyo SV-2100S4, Japan). The surface roughness values were noted for each ground sample in three distinct locations. The roughness measurement specifications were 4 mm transverse length, 0.8 mm cut-off length, and 0.02 mm/s traverse speed. A cantilever-type probe with a 2 µm diamond stylus attached is used to get the ground surface profiles (refer to Fig. 3.9). The decreased surface roughness enhances the ground sample's physical, mechanical, and tribological qualities. Several standard roughness parameters, i.e., R_a, R_q, and R_z, describe quantitive information of surface quality. Generally, surface roughness is influenced by the wheel wear rate, adhesion of microchips and grits, and lubricating conditions during the grinding [208].

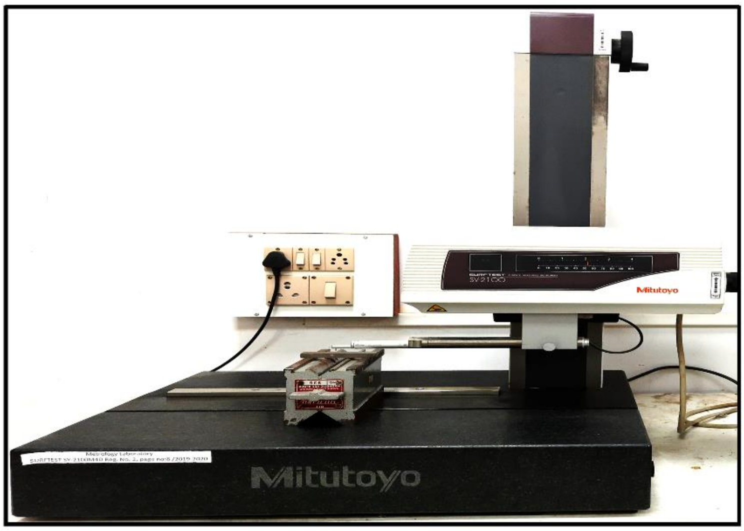
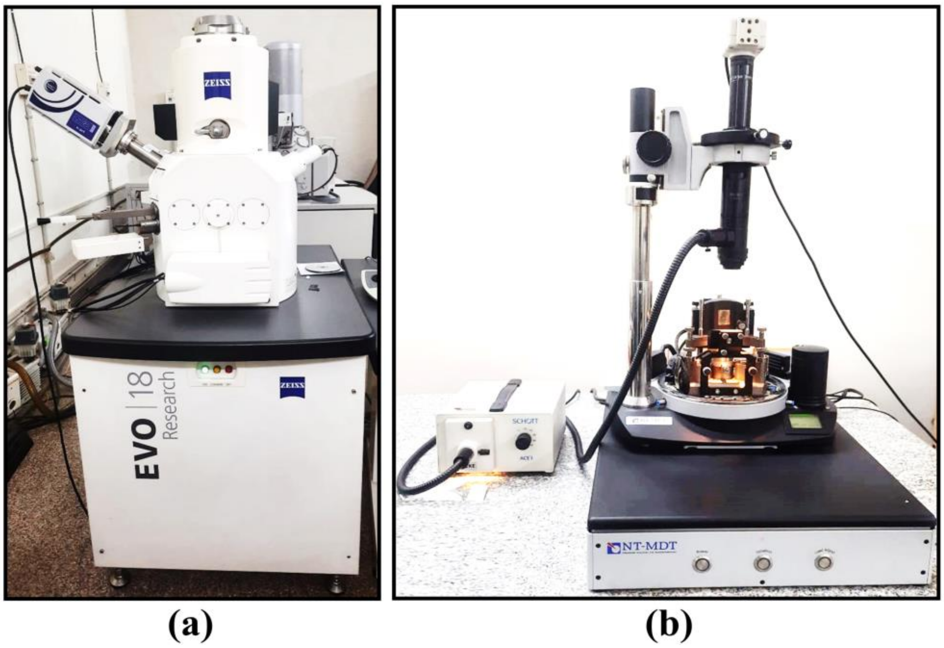


Fig. 3.9: Surface roughness measurement arrangement for ground sample.

3.5.4 Surface modifications

The surface modification section covered the surface topography of ground samples. Surface topography is a crucial evaluation index that helps determine the surface integrity of machined specimens. When it comes to directly evaluating the surface integrity of ground samples, the scanning electron microscope (SEM), energy-dispersive X-ray spectroscopy (EDS) and the atomic force microscope (AFM) are generally considered to be the most effective methods. High-resolution images of the surface of ground samples can be obtained by SEM. Using SEM, researchers can examine a ground sample's topography and composition in detail. The SEM uses an electron beam to scan the surface of the ground sample and generates images with high magnification and resolution. The SEM can also provide information on the elemental composition of the ground sample by using an EDS detector. In EDS, a sample is bombarded with a concentrated electron beam to produce X-rays, which are then detected and examined. Each element in the sample results in a unique set of X-ray peaks that can be used to determine which elements are present. These peaks' amplitudes reveal details about the relative concentrations of the elements. The present research performed SEM and EDS analysis on the scanning electron microscope equipment (Zeiss EVO-18-Research, Germany) for a 10 × 10 mm^2 dimension of ground sample. These samples were cut by Wire Cut EDM Machine (Series-Ex 4032C, Medha Enterprises, India). On the other hand, chip morphology is also analyzed by SEM technique which is essential for the grindability of the work material. It was also performed on the SEM instruments (refer to Fig. 3.10 (a)).



(a) (b)

Fig. 3.10: (a) Scanning electron microscope (SEM); (b) Atomic force microscope (AFM).

Furthermore, for the investigation of three-dimensional (3D) surface topography with nano-level surface roughness parameters such as average roughness (S_a), root mean square roughness (S_q), and area peak to valley height (S_t), AFM instrument (NT-MDT NTEGRA, Russia) was used (refer to Fig. 3.10 (b)). Bearing area curve analysis was also done by obtaining outcomes from AFM instruments. This analysis described the surface characteristics of the ground surface.

3.5.5 Metallographic and microhardness analyses

Metallographic and microhardness investigations are fundamental techniques for examining the microstructure and mechanical characteristics of metals and alloys. Each ground sample was cut perpendicular to the grinding marks for the metallographic and microhardness analysis. A Wire Cut EDM Machine (Medha Enterprises, Series-Ex 4032C, India) was utilized to cut the ground sample in size of 10 × 10 mm^2. The typical metallographic method (ASTM E3-11) was followed to prepare the samples. The cross-sectional area of the samples was finally hot-molded in mount press (Chennai Metco Pvt. Ltd., India) by Bakelite particles. After completing the process in mount press, hot molded specimens were rubbed using various emery papers (grades: 180, 320, 600, 800, 1000, 1200 and 1500) and then smoothened like a mirror finish by a diamond-polishing process at the polishing device. Further, the clean sample was etched with 5% nital solution for about 15 s. Nital solution was prepared with 95 ml methanol and 5 ml nitric acid in a measuring beaker to make 100 ml nital solution. Finally, the etched sample was inspected below the light optical microscope (Dewinter, Classical PL, India) (refer to Fig. 3.11 (a)).

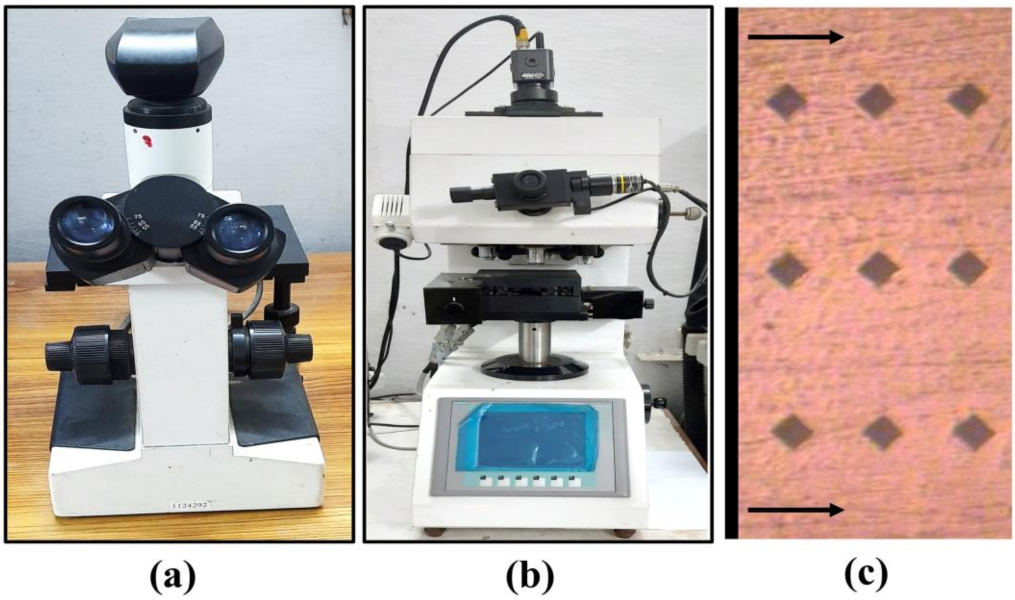


Fig. 3.11: (a) Optical microscope; (b) Microhardness tester; (c) Vickers indentations taken at different locations.

After that, the variation in the hardness along a transverse section of the ground sample was measured by a Vickers microhardness tester (Micro-Mach Technologies, India) with an applied load of 100 g for 10 s dwell time. Fig. 3.11 (b) shows the microhardness tester. Vickers indentation started 30 μm away from the edge of the ground surface and reached up to 430 μm at three distinct positions, as depicted in Fig. 3.11 (c). The distance between the two subsequent indentations was 50 μm. For analysis, the microhardness average value was taken into account.