

CHAPTER – 2

Materials

and

Experimental Details

This chapter provides details on materials selection, synthesis, processing, and characterization techniques used for carrying out the investigations elaborated in Chapters 3, 4, and 5. Al-based quasicrystalline alloys and composites synthesis and processing, quasicrystal and non-equiatomic HEAs reinforced AMCs through various non-equilibrium routes notably vacuum induction melting, mechanical milling via high-energy ball milling, and spark plasma sintering is described in details. The vacuum induction melting was used for the synthesis of as-cast Al-based periodic and aperiodic alloys used in the present work. These Al-based based quasicrystalline composites and AMCs were processed through mechanical milling for uniform dispersion of reinforcement and sintered by SPS, HP and pressure-less sintering. The effect of milling duration, the role of reinforcement on the structural transformation, grain refinement, morphology, and mechanical properties of quasicrystalline and Al matrix composite powder and sintered pellets were established through various characterization techniques. The structural change of quasicrystalline composite and AMCs were investigated through X-ray diffraction (XRD) and transmission electron microscopy (TEM). The morphology, microstructural features and elemental composition of milled powders and sintered materials were discerned through optical microscopy (OM), scanning electron microscopy (SEM) equipped with X-ray energy dispersive spectroscopy (EDS) and TEM-EDS techniques. The phase evolution of these composites during heating was corroborated through differential scanning calorimetry (DSC) and in-situ/ ex-situ XRD. The density of the sintered composite were also evaluated. The indentation hardness of mechanically milled powders and sintered composites were measured through microhardness and nanohardness techniques. Additionally, the mechanical properties were also discerned through compressive testing for SPSed materials at room temperature. The details of the alloys synthesis, processing of composites, and its

structural, thermal, and mechanical characterization have been described in detail in the following sections.

2.1 Materials designation

In the present work, mainly three types of composites have been synthesized as mentioned below:

- Al-Cu-Fe quasicrystalline matrix composite reinforced with Sn particles
- AA 6082 Al matrix composite reinforced with Al-Cu-Fe quasicrystals
- AA 6082 Al matrix composite reinforced with non-equiatomic Al-Si-Cr-Mn-Fe-Ni-Cu HEA

The Al-Cu-Fe quasicrystalline alloy has a nominal composition of $\text{Al}_{62.5}\text{Cu}_{25}\text{Fe}_{12.5}$ (at%) and is designated as IQC. The designation of composite synthesized in the present investigation are described in Table 2.1.

Table 2. 1: Designation of composite synthesized in the present investigation.

Matrix	Reinforcement	Volume fraction of reinforcement	Designation
$\text{Al}_{62.5}\text{Cu}_{25}\text{Fe}_{12.5}$ (at%)	Sn	10	IQC-10Sn
		20	IQC-20Sn
		30	IQC-30Sn
AA 6082 Al	$\text{Al}_{62.5}\text{Cu}_{25}\text{Fe}_{12.5}$ (at%)	0	Al-0IQC
		10	Al-10IQC
		20	Al-20IQC
		30	Al-30IQC
		40	Al-40IQC

AA 6082 Al	Al ₄₀ (SiCrMnFeNiCu) ₆₀ (at%)	10	Al-10HEA
		20	Al-20HEA
		30	Al-30HEA

2.2 Materials synthesis

The metallic shots of Al, Si, Cr, Mn, Fe, Ni, and Cu (purity $\geq 99\%$; Alfa Aesar India Pvt. Ltd.) were used for the synthesis of Al-Cu-Fe based IQC alloy and non-equiatomic AlSiCrMnFeNiCu HEA alloy. The elemental powder of Sn having a purity $\geq 99.5\%$ was used for the synthesis of IQC-Sn composites. The physical properties of metallic shots and elemental powder used in the present investigation are briefly mentioned in Table 2.2.

Table 2. 2: Physical properties of elements used for the synthesis of alloys and composites.

Elements	Al	Si	Cr	Mn	Fe	Ni	Cu	Sn
Crystal structure	FCC	DC	BCC	Cubic	BCC	FCC	FCC	BCT
Density (g.cm⁻³)	2.70	2.33	7.19	7.43	7.86	8.90	8.96	7.31
Melting point (°C)	660	1414	1907	1246	1538	1455	1084	231

The IQC and non – equiatomic HEA alloys used in the present investigation were prepared by vacuum induction melting. The AA 6082 Al alloys used for AMCs were prepared by gas atomization techniques.

2.2.1 Vacuum induction melting

The Al-Cu-Fe IQC and non – equiatomic AlSiCrMnFeNiCu HEA alloys having a nominal composition of Al_{62.5}Cu₂₅Fe_{12.5} (at%) and Al₄₀(SiCrMnFeNiCu)₆₀ (at%) were synthesized by vacuum induction melting (VIM), respectively. The metal shots of Al, Cu, and Fe (for IQC alloy) and Al, Si, Cr, Mn, Fe, Ni & Cu (for non – equiatomic HEA)

weighing 1 kg was placed in an alumina crucible inside the chamber of vacuum induction melting. The alumina crucible containing the metal shots were surrounded by water-cooled copper coils for supplying AC current. The chamber was backfilled with Argon gas to avoid the evaporation of metals due to the difference in the melting point of constituent elements. The copper coil was connected with the solid-state RF generator for supplying AC current. The metal shots placed inside the alumina crucible experiences a varying magnetic field as well as eddy current. The flow of the eddy current inside the metal leads to its resistive heating and may be varied as a function of RF power. After the melting and homogenous mixing of metal in the alumina crucible, the molten alloy was poured into the water-cooled copper crucible. During the VIM, the temperature of molten metal was monitored with the help of an optical pyrometer. The Al-Cu-Fe IQC and AlSiCrMnFeNiCu HEA alloys were poured at a temperature of ~ 1400 °C (1673 K). The as-cast IQC and non – equiatomic HEA alloys were having a density of 4.18 g.cm^{-3} and 5.08 g.cm^{-3} , respectively.

The as-cast alloy was having icosahedral quasicrystalline (IQC) and B2-type Al (Cu, Fe) phases in the ternary Al-Cu-Fe system. For synthesizing stable IQC phase in Al-Cu-Fe alloys, suitable annealing treatment was given to the as-cast alloy. The as-cast alloy was annealed at 800 °C (1073 K) for 10 h in a resistance furnace with a heating rate of 5 °C/min. After completion of annealing treatment, the alloy was water quenched to arrest the high-temperature phases. This annealing treatment followed by quenching leads to forming a stable face-centered ordered IQC phase in the Al-Cu-Fe alloys without any prominent signature of B2-type Al (Cu, Fe) or $\text{Al}_{13}\text{Fe}_4$ phases.

2.2.2 Gas atomized Al alloy

The age-hardenable AA 6082 Al alloy powders were produced by the process of gas atomization having purity $\geq 99\%$, particle size $< 75 \mu\text{m}$ was supplied by Kemphasol

Ltd., India. The chemical composition AA 6082 Al alloy powders are mentioned in Table 2.3.

Table 2. 3: Chemical composition of gas atomized 6082 Al alloy powder used for AMCs.

Elemental composition (in wt %)							
Si	Mg	Mn	Fe	Cr	Cu	Zn	Al
1.20	0.78	0.50	0.33	0.14	0.08	0.05	Bal.

2.3 Composite powder production

In this work, nanocomposites of Al-Cu-Fe IQC reinforced with Sn particles, and AA 6082 Al matrix composite reinforced Al-Cu-Fe IQC particles, and Al matrix composite reinforced with HEA particles were prepared by mechanical milling. For preparing powders of IQC-Sn, Al-IQC, and Al-HEA nanocomposites, the matrix and reinforcement were weighed in the desired proportion. The weight fraction of the matrix and reinforcement were calculated as per the rule of mixture [212], as mentioned in equation 2.1.

$$V(\text{reinforcement}) = \frac{\frac{w(\text{reinforcement})}{\rho(\text{reinforcement})}}{\frac{w(\text{matrix})}{\rho(\text{matrix})} + \frac{w(\text{reinforcement})}{\rho(\text{reinforcement})}} \dots \dots \dots (2.1)$$

Where, V (reinforcement) = volume fraction of reinforcement

W (reinforcement) = weight fraction of reinforcement

W (matrix) = weight fraction of matrix

ρ (reinforcement) = Density of reinforcement (g.cm^{-3})

ρ (matrix) = Density of matrix (g.cm^{-3})

The weight fraction of matrix and reinforcement in IQC-Sn, Al-IQC and Al-HEA nanocomposites were calculated in accordance to equation 2.1 and are mentioned in Table 2.4.

Table 2. 4: Weight fraction of matrix and reinforcement used for the synthesis of nanocomposites.

Designation	V_(reinforcement)	W_(reinforcement)	W_(matrix)
IQC-10Sn	10	0.16	0.84
IQC-20Sn	20	0.31	0.69
IQC-30Sn	30	0.43	0.57
Al-10IQC	10	0.15	0.85
Al-20IQC	20	0.28	0.72
Al-30IQC	30	0.40	0.60
Al-40IQC	40	0.51	0.49
Al-10HEA	10	0.17	0.83
Al-20HEA	20	0.32	0.68
Al-30HEA	30	0.45	0.55

For preparing the smaller size particles of as-cast Al-Cu-Fe IQC and non – equiatomic AlSiCrMnFeNiCu HEA alloys, a vibratory ball mill was used for crushing and fragmentation. This was used as starting materials for synthesizing the nanocomposite powders through mechanical milling in a high-energy ball mill.

2.3.1 Vibratory ball milling

Vibratory ball milling was used for crushing and fragmenting the as-cast & annealed Al-Cu-Fe IQC and as-cast non – equiatomic AlSiCrMnFeNiCu HEA into powder particles having size $\leq 100 \mu\text{m}$. The schematic of the vibratory ball mill (KC-0, Tau Instrument) setup used for the present work is illustrated in Figure 2.1.

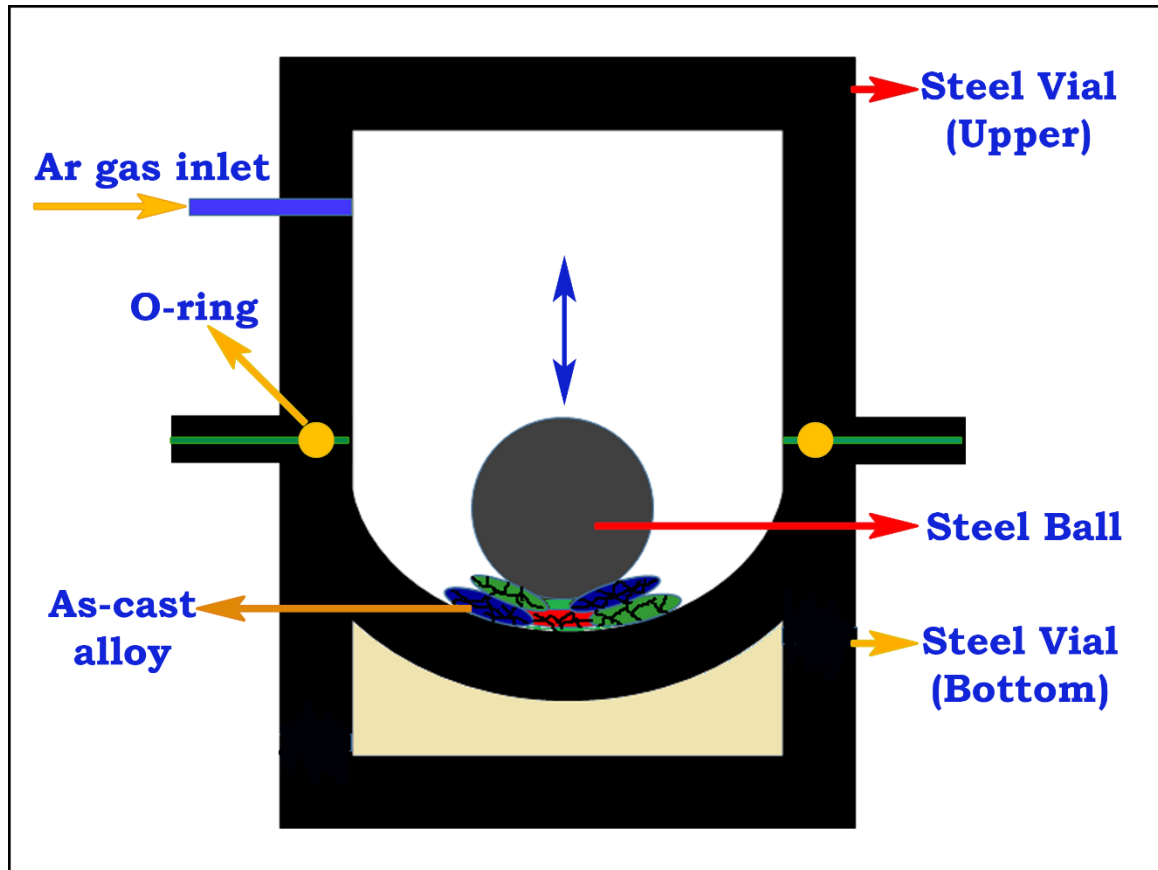


Figure 2. 1: Schematic of KC-0 cryobox/vial used for fragmentation of as-cast alloys.

The samples are loaded into the cryobox (vial) made up of steel, in which as-cast samples were loaded with a 65 mm diameter stainless steel ball. After loading the sample and tightening the vial, the upper half of the vial was connected to Argon gas cylinder, and its constant flow was maintained to avoid any contamination during vibratory milling. The cryobox/ vial was vibrated for 60 s with a pause of 10s to prevent the formation of a dead zone during the process, and this was continued for 30 min with a ball to powder ratio (BPR) of 100:1. Due to the high impact during vibratory ball milling, the as-cast Al-Cu-Fe IQC and non – equiatomic AlSiCrMnFeNiCu HEA were fragmented into smaller size powder particles and was directly used for mechanical milling to synthesize IQC-Sn, Al-IQC, and Al-HEA nanocomposite powders through high energy ball milling.

2.3.2 High energy ball milling

The powder particles of IQC & Sn, Al & IQC and Al & non – equiatomic HEA were taken in the desired proportion, as mentioned in Table 2.4. This was fed (~30-40 g) as a starting material for synthesizing IQC-Sn, Al-IQC, and Al-HEA nanocomposite powders through mechanical milling. The mechanical milling was carried out in a high-energy planetary ball mill (PM 400 & PM 400/2; Make: Restsch, Germany) at 200 rpm with a BPR of 10:1. The composite powders were milled in 250 ml tungsten carbide (WC) vials with WC balls of 10 mm diameter. In the present work, toluene was used as a process control reagent to avoid oxidation during milling. The process parameters for milling are mentioned in Table 2.5.

Table 2. 5: Protocol for mechanical milling of nanocomposite powders.

Sample designation	Milling Duration	Sample withdrawal duration	Running protocol		Vials and balls	Milling medium	Process control reagent
			Mill running duration	Mill rest duration			
IQC-Sn	40 h	10 h, 20 h, 30 h & 40 h	30 min	15 min	WC	Wet	Toluene
Al-IQC	50 h	10 h, 20 h, 30 h, 40 h & 50 h	30 min	15 min	WC	Wet	Toluene
Al-HEA	50 h	10 h, 20 h, 30 h, 40 h & 50 h	30 min	15 min	WC	Wet	Toluene

During mechanical milling, samples were withdrawn from the vials after every 10 h to ascertain structural transformation. Intermittently the high-energy planetary ball mill was stopped for 15 min after every 30 min of mechanical milling to avoid overheating during mechanical milling.

2.4 Powder compaction

The mechanically milled powders of Sn reinforced Al-Cu-Fe IQC matrix nanocomposite, and Al-Cu-Fe IQC reinforced AA 6082 Al matrix nanocomposite were consolidated by spark plasma sintering (SPS). Additionally, the IQC-10Sn nanocomposite powders were sintered using hot pressing.

2.4.1 Spark plasma sintering

The densification of nanocomposite powders of IQC-Sn and Al-IQC were carried out using spark plasma sintering (Model: 25-10, 25 Ton load, Current: 10,000 A; Make: Thermal Technology LLC, USA). The parameters for densification used for fabricating the bulk samples of IQC-Sn and Al-IQC composite through SPS are mentioned in Table 2.6.

The nanocomposite powders of IQC-Sn and Al-IQC were loaded in a graphite die after putting a 500 μm thick graphite foil at the inner periphery of the die surrounding the powder and in between the punches & powder. The graphite foil was used to easily withdraw samples from graphite die after densification as the parameters mentioned in Table 2.6. The temperature during spark plasma sintering was measured using a k-type thermocouple. The thermocouple was inserted at a distance of 0.002 mm from the inner wall of the graphite crucible to minimize the temperature difference between die and samples. The graphite die was placed between the electrode (for applying pulsed current) in the SPS chamber under a vacuum of 10^{-3} Torr. During the spark plasma sintering process, Ar gas was purged in the SPS chamber at a flow rate of 5.0 l/min. The spark plasma sintered samples of IQC-Sn and Al-IQC composites have a thickness of 8-10 mm.

Table 2. 6: Processing parameters of IQC-Sn and Al-IQC composites during SPS.

Designation	Temperature (°C)	Pressure (in MPa)	Holding time (in min)	Heating rate (°C/min)	Diameter of pellets (in mm)
IQC-10Sn	800	50	15	100	15
IQC-20Sn	800	50	15	100	15
IQC-30Sn	800	50	15	100	15
Al-0IQC	300	500	30	100	20
Al-10IQC	300	500	30	100	20
Al-20IQC	300	500	30	100	20
Al-30IQC	300	500	30	100	20
Al-40IQC	450, 550	50	5	100	20

For spark plasma sintering at high pressure (500 MPa) and comparatively low temperature (300 °C), tungsten carbide (WC) die and punches were used. In this case, also for the hassle-free ejection of samples, graphite foils were used at the interfaces of nanocomposite powders and die/ punch. The WC dies, and punches were used for high-pressure sintering, as graphite die cannot sustain such high pressures.

2.4.2 Hot pressing

The IQC-10Sn nanocomposite powders was additionally sintered through hot pressing. The die made up of H13 die steel of 10 mm diameter was fabricated for this particular purpose. The die cavity, bottom, and upper punch were coated with a thin layer of boron nitride (BN) to avoid powder sticking to the die during sintering. The nanocomposite powder of IQC-10Sn was (5-6 g) was placed inside the die and was kept in the hydraulic press with the resistive heating arrangement (100-Ton capacity; Crane-Bel

International Pvt. Ltd.). The resistance heating method was used for obtaining the desired temperature during hot pressing. The sample was hot-pressed at 650 °C (923 K) at a pressure of 650 MPa for 15 min in an ambient atmosphere. The sintered pellets were having a thickness of 4-5 mm.

2.5 Structural Characterization

In order to investigate the structural transformation during mechanical milling, to ascertain the size, shape and distribution of phase, microstructural features, and phase composition of nanocomposite powders as well as sintered composite, several analytical methods were used. The structure of IQC-Sn, Al-IQC and Al-HEA was characterized by X-ray diffraction (XRD) and transmission electron microscopy (TEM). The microstructure of these composites were discerned through optical microscopy (OM), scanning electron microscopy and TEM. For evaluating the elemental composition of composites, analytical techniques like SEM-EDS (X-ray energy dispersive spectroscopy) and TEM-EDS were used.

2.5.1 X-ray diffraction

The standard X-ray diffraction (XRD) instrument (EMPYREAN, PANalytical) having Co-K α radiation ($\lambda=0.179$ nm) at an operating voltage of 40 kV and 40 mA was used for evaluating the phases in nanocomposite powders and spark plasma sintered samples. The samples were scanned in a 2θ range of 20° to 120° (for most of the samples) using a step size of 0.02° with a scan rate of 5°.min⁻¹. The phase transformation in Al-HEA nanocomposite as a function of temperature were investigated through in-situ XRD (Rigaku Smart Lab) having Cu-K α radiation ($\lambda=0.154$ nm) at an operating voltage of 45 kV and 200 mA. The samples were scanned in a 2θ range of 20° to 80° using a step size of 0.02° with a scan rate of 5°.min⁻¹. The phases evolved in as-cast alloys, milled composite

and sintered pellets were analysed with the help of "International Centre for Diffraction Data (ICDD)" PDF2 database.

In the present investigation, Williamson–Hall method [354] was used for calculating the crystallite size (t) and lattice strain (ϵ) by equation 2.2 and illustrated in Figure 2.2.

$$\beta \cdot \cos\theta = \frac{0.9\lambda}{t} + 4\epsilon \cdot \sin\theta \dots\dots\dots (2.2)$$

where β = total broadening due to crystallite size and lattice strain

λ = wavelength of radiation used for XRD

θ = Bragg angle

The Pseudo – Voigt function was used for profile fitting of XRD peaks for computing the peak broadening. The total broadening was calculated after subtracting the standard Si sample's broadening for omitting the instrumental broadening.

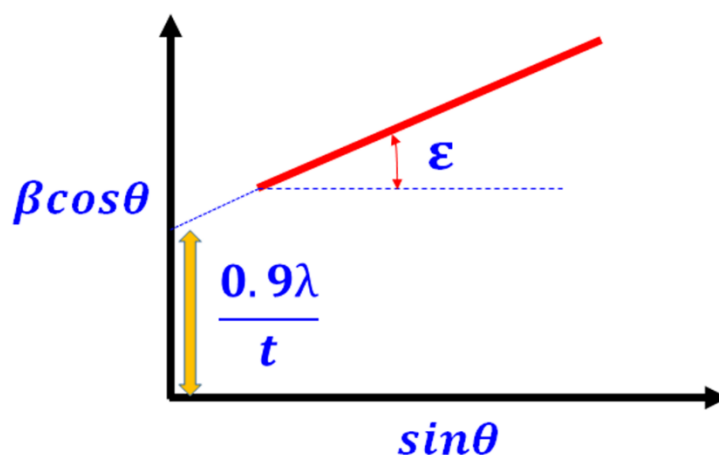


Figure 2. 2: Williamson – Hall plot for computing crystallite size and lattice strain.

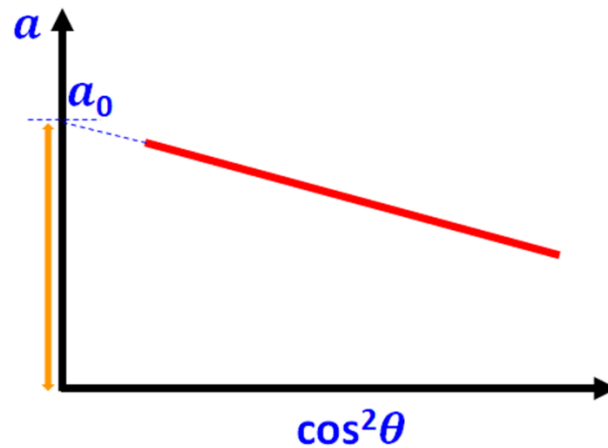


Figure 2. 3: Precision lattice parameter by Bradley – Jay method.

The lattice parameter (a) of the phases evolved during the mechanical milling of nanocomposites were evaluated by computing the precision lattice parameter (a_0). The powders precision lattice parameter was calculated with the help of the Bradley – Jay method [355], as illustrated in Figure 2.3.

The dislocation density of Al-HEA nanocomposite powders were calculated using the expression given below [356]:

$$\text{Dislocation density, } \rho = \frac{3\sqrt{2}\pi\langle\varepsilon^2\rangle^{1/2}}{tb} \dots\dots\dots(2.3)$$

Where 't' and ε are the crystallite size and lattice strain, b is the Burgers vector of FCC

metals [$b = \frac{a\sqrt{2}}{2}$; a = lattice parameter (Å)].

2.5.2 Optical microscopy

The optical metallographic techniques used to prepare the IQC-Sn nanocomposite powders embedded in epoxy. The samples were first flattened by mechanical grinding with silicon carbide paper of 60 μm grit size to make both the faces parallel. After mechanical grinding, samples were polished on emery paper from 400 μm grit size to 2500 μm grit size. The final polishing of the samples was done on a TriDent and ChemoMet cloth

(trademark of Buehler) with diamond suspension (3 μm) and colloidal silica (0.02 μm), respectively. These samples were etched with Keller's reagent after completion of final polishing for 5 – 10 s. The etched samples' microstructure was captured using an optical microscope (Imager.M1m, AX 10 model; Make – Carl Zeiss) at various magnification.

2.5.3 Scanning electron microscopy

The scanning electron microscopy is a useful instrument for characterizing the microstructural details from a length scale ranging from millimeters to nanometre. In general, SEM operates in two types of modes i.e., secondary electron (SE) mode and backscattered electron (BSE) mode. The SE mode is used for topographical information of samples; on the other hand, BSE mode is used for detecting the contrast between the areas of different chemical composition.

In the present work, characterization of the nanocomposite powders and sintered pellets was studied through EVO 18 (Carl Zeiss) SEM and high-resolution FESEM (Quanta 200F & NOVA NanoSEM 450) with FEG source. For elemental analysis, the SEM has equipped with an X-ray energy dispersive spectroscopy (EDS) detector. The sample preparation of SEM is similar to optical microscopy, as described in section 2.5.2. The powder samples embedded in epoxy were coated with Au (Gold) in desk sputter coater to avoid charging samples during SEM investigation. The nanocomposite powder samples were sprinkled on the carbon tape attached to the SEM stub of its imaging.

2.5.4 Transmission electron microscopy

To confirm the structure obtained by XRD experiments and to investigate fine microstructural features, transmission electron microscopy (TEM) was used. TEM studies were performed using a FEI Tecnai G2 T20 S-twin microscope equipped with the high-angle annular dark-field (HAADF) and EDS detectors operating at 200 kV.

The powder samples were dispersed in methanol/ ethanol, followed by ultrasonication (for 15 – 20 min) for avoiding the accumulation of powder particles. These suspended powder particles in methanol/ ethanol (1 – 2 drop) dropped cast onto a 3 mm copper grid (200 mesh size) and dried with the help of an infrared lamp for 30 min. The bulk samples of Al-40IQC composite samples were prepared by sectioning the spark plasma sintered pellet into 500 μm thick foil. This foil was mechanically polished using SiC papers of 400 – 2500 μm grit size for reducing its thickness to ~ 70 μm . The 70 μm thick foil was punched to 3 mm diameter foil with disk puncher (from GATAN) followed by electrolytic thinning for preparing electron transparent samples for TEM. The electrolytic thinning was done using twin-jet electro-polisher (Model I20, FISCHIONE) with the electrolyte of 20% HNO_3 and 80% CH_3OH (volume fraction) at the cryogenic temperature of -20 $^\circ\text{C}$ (253 K) was used for preparing the electron transparent samples for TEM investigations.

2.6 Thermal stability

The thermal stability of nanocomposite powders of IQC-Sn and Al-HEA were discerned through differential scanning calorimetry (DSC). The phase evolution events corresponding to DSC thermogram were confirmed through ex-situ XRD of IQC-Sn nanocomposite annealed samples and in-situ XRD of Al-HEA nanocomposite powders.

2.6.1 Differential scanning calorimetry

The thermal analysis of mechanically milled nanocomposite powder particles was done using a differential scanning calorimeter (DSC 404 F3 Pegasus, NETZSCH, Germany) under a continuous nitrogen flow gas. The Alumina crucible was used as a reference and a sample holder crucible. They were charged with ~ 20 to 30 g of mechanically milled powder particles. The calibration of DSC instrument was done using

indium, silver, and nickel with temperature error well within the permissible range. The DSC of samples were performed at a various heating rate to confirm the phase transformation events and compute activation energy associated with those events.

2.6.2 Annealing treatment

The IQC-Sn nanocomposite powders mechanically milled for 40 h were annealed from 300 °C to 800 °C with a temperature interval of 100 °C for 10 h. The powder samples ~10 g was encapsulated in a quartz tube backfilled with Argon gas. These encapsulated powders were annealed at different temperatures in a muffle furnace with a heating rate of 5°/min and furnace cooled to room temperature. The phases in IQC-Sn annealed samples were confirmed through ex-situ XRD, and these were further co-related to heating events of DSC thermogram.

2.7 Physical and mechanical properties

2.7.1 Density measurements

The Archimedes principle was used to measure the density of sintered pellets of IQC-Sn and Al-IQC. The density of sintered samples $\rho_{(sample)}$ was calculated by equation 2.4.

$$\rho(sample) = \frac{m(air)[\rho(liquid)-\rho(air)]}{m(air)-m(liquid)} \dots\dots\dots (2.4)$$

Where $\rho_{(liquid)}$ is the density of the liquid medium, $\rho_{(air)}$ is the density of air, $m_{(air)}$ is sample weight in ambiance, and $m_{(liquid)}$ is the weight of the sample immersed in the liquid medium. The samples were first measured in air and then in a liquid medium using an electronic weighing balance (Model No- CAH-503, CONTECH Instrument Ltd., INDIA) equipped with a density measurement module. Further, the relative density of the samples was calculated by

$$\text{Relative density (\%)} = \frac{\rho(\text{experimental})}{\rho(\text{theoretical})} * 100 \dots \dots \dots (2.5)$$

Where $\rho_{(\text{experimental})}$ is the observed density of sintered pellets computed by equation 2.4; $\rho_{(\text{theoretical})}$ is the theoretical density calculated by the rule of mixtures [212].

2.7.2 Hardness

The hardness of as-cast alloys, mechanically milled nanocomposite powders and sintered pellets were determined using both microhardness tester and instrumented indentation testers. The nanocomposite powders were embedded in Epoxy (EpoxiCure2 resin and hardener; Buehler), having an epoxy resin to hardener ratio of 4:1. The customized moulds coated with the release agent were filled with a 2 mm thick layer of mechanically milled powder particles. The epoxy resin and hardener mixed in the desired proportion was poured into the mould and allowed to settle at room temperature for 6 h. The powder samples and sintered pellets were mirror-polished as per the procedure described in section 2.5.2.

The microhardness of IQC-Sn powder and hot-pressed samples were measured using a microhardness tester (HMV-2; Make: Shimadzu Corporation, Japan). The microhardness powder samples embedded in epoxy was measured at a load of 100 mN with a dwell time of 10 s. The nanohardness measurements of the individual IQC-Sn nanocomposite powder particles were carried out using standard Berkovich nanoindenter (TI950, Hysitron) at a peak load of 5 mN and loading rate of 20 $\mu\text{N/s}$. The reported hardness of nanocomposite powders is at least an average of ten measurements. The microhardness of the sintered pellets was computed through an instrumented microhardness tester (MHT3; Make: Anton Paar, Switzerland) with Vickers indenter at various load. The microhardness of Al-HEA pressure-less sintered sample embedded in epoxy was measured at a load of 1000 mN with a dwell time of 20 s.

2.7.3 Compressive testing

The compressive testing of spark plasma sintered samples of IQC-Sn and Al-IQC composite was determined in order to access the compressive strength. Compressive properties of IQC-Sn and Al-IQC sintered samples were determined using a 100 kN screw-driven InstronTM, Universal Testing Machine (Model 4206). All the compressive test specimens were prepared as per the ASTM E9 specification for the cylindrical specimen. The cylindrical samples were cut into the desired geometry by wire-cut EDM machine. The sintered samples have a length of 8 mm and a diameter of 4 mm for Al-IQC composite. The IQC-Sn composite samples have a height of 6 mm and a diameter of 3 mm. All the cylindrical samples had a length to diameter ratio of 2:1. The crosshead speed for all the samples was maintained at a constant rate of 0.5 mm/min.