EFFECT OF QUENCHING MEDIA ON PROPERTIES OF ALUMINIUM-ALUMINA METAL MATRIX COMPOSITE

This thesis is submitted in the partial fulfillment of the requirement for the degree of **Bachelor of Technology**

In

METALLURGICAL AND MATERIALS ENGINEERING

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CERTIFICATE

This is to certify that the thesis entitled "Study of effects of quenching media on properties of aluminum-alumina metal matrix composite" submitted by Anuj Dash (110MM0097) and Anil Kumar Nayak (110MM0367) in partial fulfilment of the requirements for the award of BACHELOR OF TECHNOLOGY Degree in Metallurgical and Materials Engineering at the National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/ Institute for the award of any degree or diploma.

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ABSTRACT

Particle reinforced aluminium matrix composites are frequently used in various automotive and aerospace applications involving critical functions like piston rings, wing mid and inner cover etc. Here the composites are frequently subjected to extreme conditions involving large temperature differentials in a short period of time like in pistons facing an average temperature of 300 ^{0}C or in aeroplane wings facing cryogenic temperatures at high altitudes. Such conditions can deteriorate the particle matrix interface resulting in a decreased ability to resist matrix deformation or strengthen the matrix through dislocation generation due to thermal misfit between matrix and reinforcement. Hence, this phenomena has been investigated in the course of this work by quenching aluminium alumina micro and nano composites in different quenchants and studying the effects on mechanical properties as well as the microstructure. Increase in hardness was likely due to dislocation generation as was realized from the greater increase for higher vol. fration reinforcement composites, while post quench softening was likely due to decohesion and cracking of matrix and particles.

Key words: cryogenic temperature, quenching, decohesion, micro-composites, nano-composites.

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1. Introduction

Technological development is complemented by development of materials. The best technological design in aerospace or structural or automotive field is of no use in absence of appropriate materials to bear the service loads and the service conditions. Composite materials therefore represent a new endeavour in the ever advancing field of materials science to develop better materials for optimization of materials.

The American Society of Metals (ASM) defines composites as- a macroscopic combination of two or more distinct materials, having a recognizable interface between them [1]. From a restricted viewpoint, composite materials are used with respect to the materials having a continuous matrix constituent that binds together and provides form to an array of stronger and stiffer reinforcement constituent. Such a composite has a balance of structural properties that is superior to either constituent material alone.

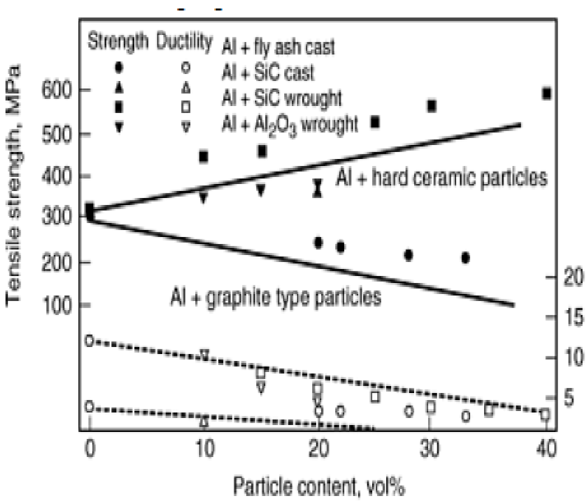
In the course of the past five decades, composites especially metal matrix composites have emerged as an important class of materials in the structural, aeronautical, automotive field, and are increasing their presence in our daily lives. Metal matrix composites are replacing conventional monolithic and alloyed materials for better mechanical properties that can be tailored to meet the specific requirement. The advantage of a MMC can only be realized on the achievement of a reasonable cost-performance relationship.

Important metal matrix materials for MMC include aluminium alloys, titanium alloys, magnesium alloys and copper alloys. Typical reinforcements can be in form of particles, whiskers or fibers. Popular materials for reinforcement are SiC, Al_2O_3, TiC, C, B_4C, SiO_2 and also W or B or high strength steel. Some these materials are also available in form of fibers or thin wires [2].

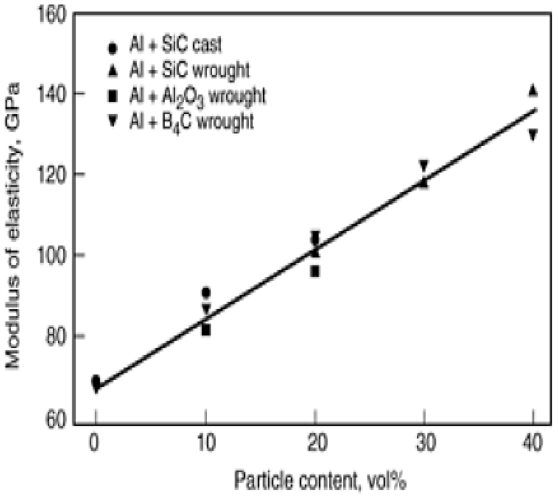
For many researchers, the term metal matrix composites is often equated with light metal matrix composites. Hence this class includes metal matrices like aluminium, magnesium, zinc, titanium etc. Aluminium alloys are the most widely used composite materials in commercial applications and in research. Examples of usage of aluminium based MMC include [2] –

- Pitch based continuous carbon fiber reinforced aluminium used for waveguide booms in Hubble telescope.
- SiCp/Al composite as a replacement of toxic beryllium in the US Trident missile.
- Short fibers of alumina or alumina + silica in diesel piston crowns
- Carbon fiber and alumina fibers reinforced aluminium alloys in cylinder liners in the Prelude model of Honda Motor Co.
- Continuous alumina fiber (Nextel 610)-reinforced Al composites in power transmission cables (e.g. 3M's Aluminum Conductor Composite Reinforced (ACCR)).
- Duralcan particulate MMCs for mountain bikes (e.g. Specialized Bicycle Co. manufactures bicycles with frames made from extruded tubes of 6061 aluminum containing about 10% alumina particles).

Incorporation of hard reinforcement particles in discontinuously reinforced Al-MMCs increases strength and stiffness due to Orowan strengthening, matrix deformation resistance etc. at the expense of ductility [3].

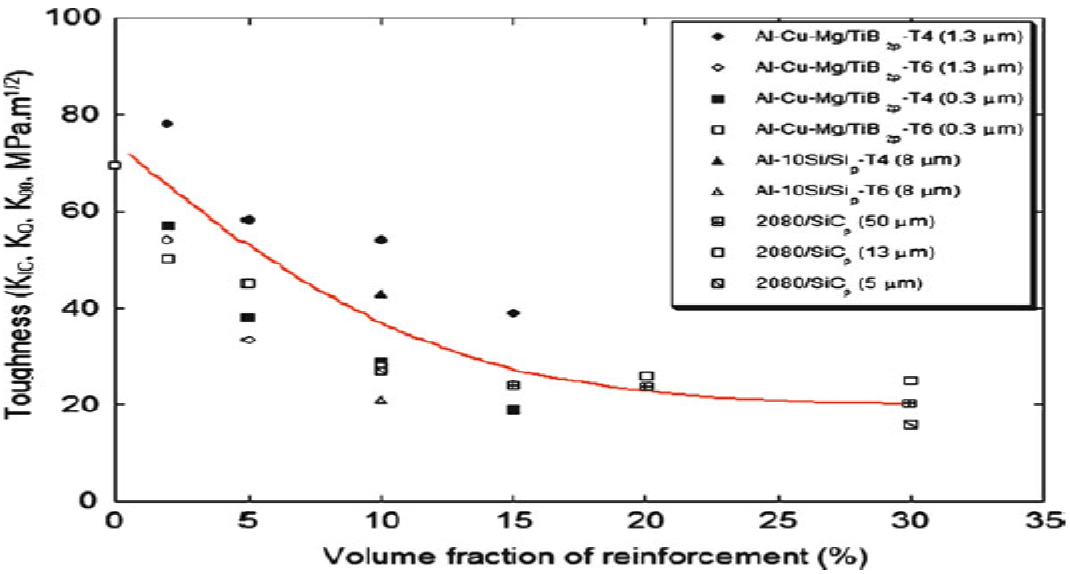


[Fig- 1(a)]



[Fig- 1(b)]

Wear resistance of an Al alloys are improved through the addition of hard particles, which improves surface hardness, or through addition of softer graphitic-type particles, which reduce the coefficient of friction between the surfaces by providing a self-lubricant. But the toughness of Al MMCs shows a decrease, with increasing volume fraction likely due to increasing stress triaxiality associated with the particles [4].



[Fig-2]

Several techniques are available for processing aluminium matrix composites. Solid state process include powder blending and consolidation, diffusion bonding, physical vapour deposition while liquid state process involve stir casting, infiltration process, spray deposition and reactive processing techniques [5]. Blending and compaction (a powder metallurgy technique) is a versatile technique for production of discontinuously reinforced aluminium matrix composites. This involves proper blending of the reinforcements and metal powders in dry or liquid suspension process, followed by cold compaction. The green samples are thereafter subjected to high temperature consolidation by hot isostatic pressing, extrusion, sintering, spark plasma sintering etc. In the simplest technique of furnace sintering, the samples are sintered at a high homologous temperature for an appropriate period of time to eliminate porosities and densify the matrix.

Mechanical properties of aluminium matrix composites depend on, apart from other factors the residual thermal stresses in the matrix. Thermal stresses can be generated by quenching from high temperature. Quenching can affect the aluminium alumina composite either by presence of quenched in vacancies, or by increase in dislocation densities through CTE mismatch between Al_2O_3 and Al matrix at interface or by dissolution of reaction products or impurities in solid state. The later two processes can increase the hardness/strength after quenching. Hence, the purpose of this study has been to perform quenching of the Al MMCs with different vol. fraction of reinforcement in different quenchants and study the effect on the microstructure and mechanical properties of the quenched samples.

1.1 Justification of the project work –

- Particle reinforced aluminium matrix composites are frequently used as a substitute of Ni cast iron in pistons of diesel or petrol based internal combustion engines to reduce weight and improve wear properties along with the ability to bear higher cylinder pressure. Under such conditions, the composites are subjected to extreme temperature variations within a very short period. This may affect reliability of the composite under critical conditions.
- Parts of the wings (inner and mid wing covers) of civilian and military aircrafts are made of particle reinforced aluminium composites. Such parts are exposed to below freezing temperatures at high altitudes and again the ambient temperatures when on ground. Such thermal cycling can affect the properties and strength of the composites.
- Parts of missile and space vehicle bodies made of particle reinforced composites are likely to face temperatures ranging from cryogenic when cruising at extremely high altitudes, to very high temperatures during re- entry. Repeated thermal cycling in re-entry vehicles may deteriorate the properties of such composites.
- Strengthening of aluminium composites by quench hardening can provide a way to improve strength, while using lesser amount of reinforcement material, provided properties remain reliable after quenching, thereby economising the composites.

2. Literature survey

2.1 Metal matrix composites:

Metal matrix composites have been of great interest in recent years due to excellent combination of properties including improved specific stiffness, specific strength, abrasion resistance, creep resistance, thermal conductivity and dimensional stability besides higher functional temperature than their base metal counterparts [5]. A simple classification of MMCs is [2] –

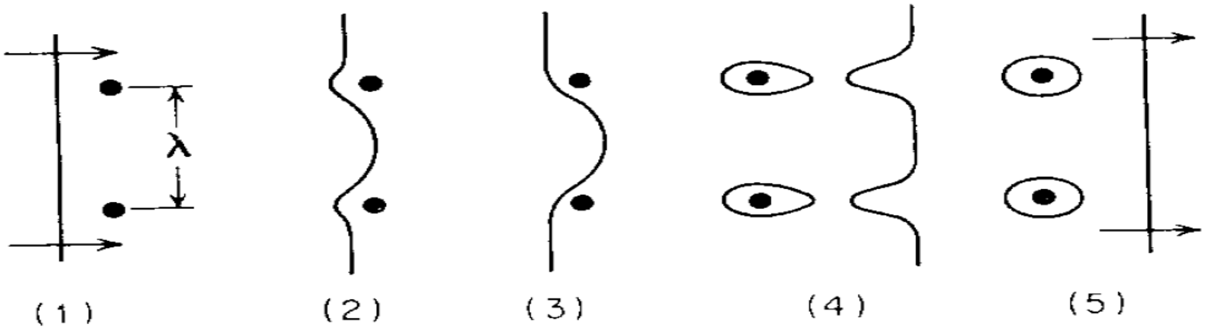
- Particle reinforced MMCs
- Whisker or short fibre reinforced MMC
- Continuous fibre reinforced MMC
- Sheet reinforced MMC or laminated MMC

Particle reinforced composites are mainly dispersion strengthened systems, involving hard reinforcement particles mixed with a ductile matrix and consolidated and processed by casting or powder metallurgy techniques. Dispersion strengthened systems have the advantage of resistance to grain growth and recrystallization at high temperatures. A critical parameter of dispersion of particles is the inter-particle spacing λ given by –

$$\lambda = \frac{4(1-f)r}{3f};$$

Here f and r are the volume fraction and the particle radius respectively [6].

For the case of incoherent precipitates, the yield stress depends on the shear stress required to bow a dislocation line between two particles separated by distance λ.



[Fig-3]

As shown in the above figures, stage 1 indicates the approach of a straight dislocation line between two particles. Thereafter, the line begins to bend in stage 2 as it cannot pass through the incoherent particles and reaches a critical curvature in stage 3. The shear stress at this stage is given by –

$$\tau_0 = \frac{Gb}{\lambda};$$

Here, G is the shear modulus and b the burger vector [6].

Incoherent particles result in strengthening by blocking dislocations as shown by the above mechanism. The resultant strengthening is given by Orowan equation –

$$\Delta\sigma = \frac{0.13Gb}{\lambda}\ln\frac{r}{b}$$

The dislocation then loops around the particles and moves forward, leaving behind a dislocation loop behind. Each successive dislocation passing through the particles leaves behind a loop of dislocation, which exert a back stress on dislocation sources. This must be overcome for additional slip to take place.

Studies [7, 8] have clearly indicated the improvement in strength and decreased wear rate on increasing the volume fraction of the reinforcements in the matrix. However, compared to particle reinforced composites, a greater improvement in strength and moduli can be achieved by incorporation of fine high strength fibres in the ductile matrix (fibre reinforced composites). This is mainly due to the fibres carrying the entire load transferred by the matrix to them. Improvement in elastic modulus and strength in unidirectional fibre strengthened composite can be estimated by –

$$E_c=E_fV_f + (1-V_f)E_m;$$

$$\sigma_{cu}=\sigma_{fu}V_f + \sigma'_m(1-V_f);$$

Here V_f is the volume fraction, E_c the composite elasticity modulus, σ'_m the matrix flow stress at fibre breaking strain. However such fibre strengthened materials have poor transverse properties and are expensive to produce and have poor recyclability. Therefore they are limited to specialized applications.

2.2 Aluminium alumina particulate composites:

Aluminium based matrix composites have been very successful as high tech material in a variety of applications. Aluminium based matrix composite application provides significant benefits like performance benefits (increased component lifetime, improved productivity), economic benefits (energy savings or lower maintenance cost) and environmental benefits (lower noise levels and fewer air-borne emissions). Such composites are more widely used than fibre reinforced composites due to their lower production costs, higher cost to benefit ratio and improved recyclability.

Alumina particle size and the volume fraction have pronounced effect on the mechanical properties of aluminium matrix composites. Addition of reinforcements to aluminium matrix results in an improvement of mechanical properties, such as hardness, strength and wears resistance. Studies [7, 8] clearly indicate improvements in strength and wear resistance obtained by increasing the volume fraction and decreasing the alumina particle size. However, the same studies also indicate reduction of elongation with increase in volume fraction and alumina size.

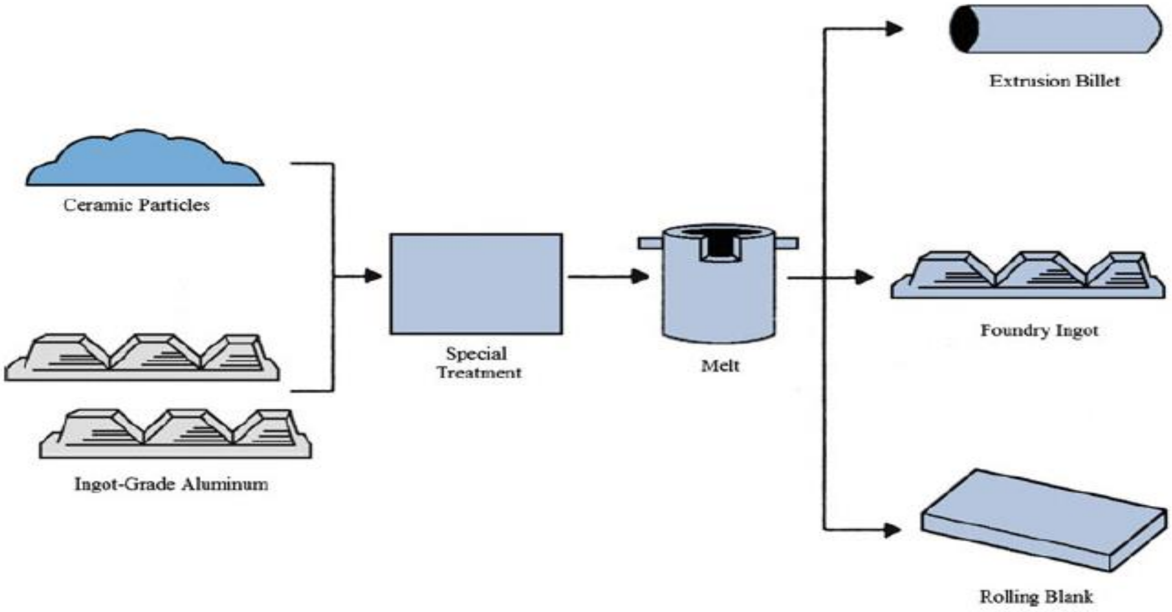
Synthesis techniques for aluminium matrix composites [4] can be divided into –

- Solid state processing - Powder blending and consolidation, physical vapour deposition and diffusion bonding.
- Liquid state processing – Stir casting, spray deposition, reactive or in situ processing and infiltration process.

part from the above techniques, several other techniques have been experimented upon for synthesis of aluminium alumina composites. For example, in order to improve the densification of composite powders, Derakhshandeh et al. used equal channel angular pressing (ECAP) of composite powders having nano alumina at 200 ^{0}C [9]. The results indicated improvement of density, micro hardness and strength due to increase in dislocation density with increasing number of passes (upto max. 4 passes for 5 and 10 vol. % alumina). Another experimental technique [10] based on friction stir processing (modification of friction stir welding) of cold sprayed aluminium alumina composite coating also yielded improved hardness due to smaller inter particle spacing. Yu et al. [11] successfully produced aluminium alumina composites using anodized and plain aluminium foils bonded by hot and cold rolling with intermediate annealing steps.

In all the above techniques, the reinforcement phase is prepared beforehand and added to the matrix phase during processing. But this creates problems like poor wettability and interfacial reactions between the matrix and reinforcements. This can be overcome through in situ processing where, the reinforcement phase develops in the matrix itself during processing. This technique involves casting of aluminium with less reactive metal oxides (e.g. CuO, MnO_2, Fe_2O_3 etc.) to produce the required Al_2O_3 in the melt itself [12-14]. However, these techniques also incorporate other reaction products into the matrix. When CuO is used, $CuAl_2$ eutectic forms at the grain boundaries [12-13] while using Fe_2O_3 incorporates Fe into the matrix [12].

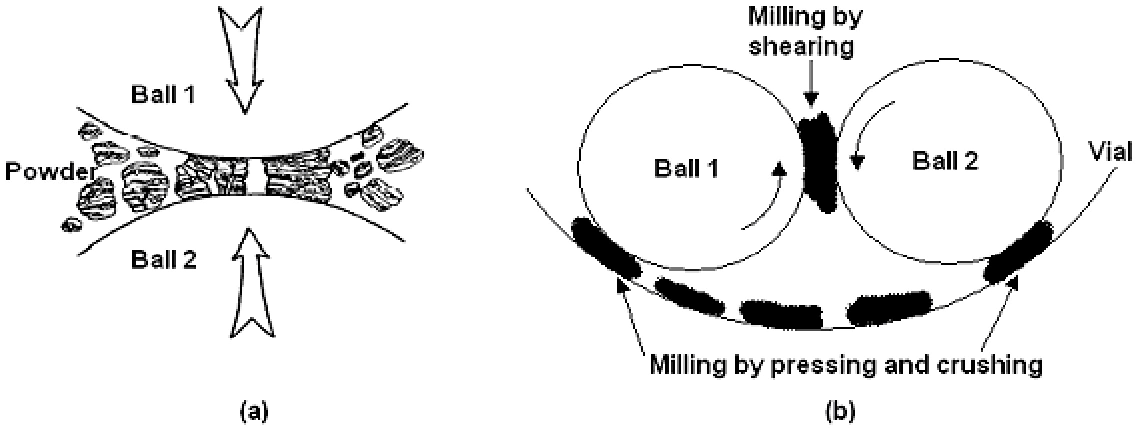
Successful commercial techniques for production of particulate aluminium composites include Duralcan process (figure below), Lanxide process (in situ) etc.



[Fig-4]

2.3 Powder metallurgy and sintering process:

Powder processing techniques are frequently used along with deformation /sintering or other consolidation techniques to produce particulate reinforced composites. This generally involves solid state blending of atomized aluminium powders and the reinforcement (alumina). The blending can be followed additionally by a milling step, to enable better distribution of the reinforcements. This is similar to high energy milling used for mechanical alloying to produce oxide dispersed superalloys. The repeated cold welding, fracture and rewelding experienced by the particles during high energy collisions between the powders and the balls (WC or steel) finally results in a steady-state particle size distribution as well as a uniform distribution of reinforcements as represented in the following figure.



[Fig-5]

The final properties depend on several milling parameters like – milling time, milling speed or energy, milling medium, milling atmosphere, ball to powder weight ratio, and the process control agent.

The blended and milled powders can then be subjected to cold powder compaction followed by sintering or be compacted and consolidated in a single step by process like Vacuum Hot Pressing (VHP), Hot Isostatic Pressing (HIP),Spark Plasma Sintering (SPS) or Severe Plastic deformation techniques (e.g. ECAP, SPT). The simplest route involves uniaxial cold compaction at a specified pressure followed by furnace sintering at a given temperature.

Sintering is a process involving a compacted assembly of loose particles metallurgically or atomically bonding into a coherent body at elevated temperature. In this process, the powders form strong bonds and incorporate any alloying element or dispersed phase. Sintering is accompanied by densification and grain growth and includes following steps sequentially [15]

- Initial particle bonding
- Neck growth
- Pore rounding
- Pore channel closure
- Densification and pore shrinkage
- Pore coarsening

The various forms of sintering include solid state sintering (powders are condensed wholly in the solid state), liquid state sintering (liquid phase present in the powder compact during the process of sintering), transient phase sintering and viscous flow sintering. Longer time and higher temperature improve bonding and densification. Studies [4] have indicated that there exists an optimum time of sintering, beyond which, hardness and strength decreases due to increase in grain size in composites. Although, the main aim of sintering is production of fully dense and fine grained microstructure, 100% densification is difficult to achieve by ordinary techniques.

2.4 Thermal stress and quench effects:

Composites experience thermal stresses associated with temperature changes during fabrication, usage etc. Such stresses are the result of the large difference of thermal expansion coefficient between the reinforcement and the matrix. The resultant residual stresses can be potentially damaging to the reliable functionality of the composite under real world situations. Such effects are magnified for quenched samples. Likely effects of quenching on the composite are –

- The influence of quenched in vacancies.
- The changes due to increased dislocation density.
- The consequences of dissolved reaction products.

Frequently, high temperature processing is likely to induce reactions between the reinforcement and the matrix to form new products [16-18]. Also contamination by oxygen etc. is likely during processing if proper precautions are not taken during synthesis [19]. Strengthening and increase in hardness can be either due to increased dislocation density arising from thermal misfit strains [19] or due to solid solution strengthening from impurity elements [20].

In general, the thermal strain developed due to misfit can be quantified as –

$\epsilon = \Delta\alpha\Delta T$;

The resultant strengthening from CTE mismatch can be expressed as –

$\Delta\sigma = \gamma G b\sqrt{\rho}$;

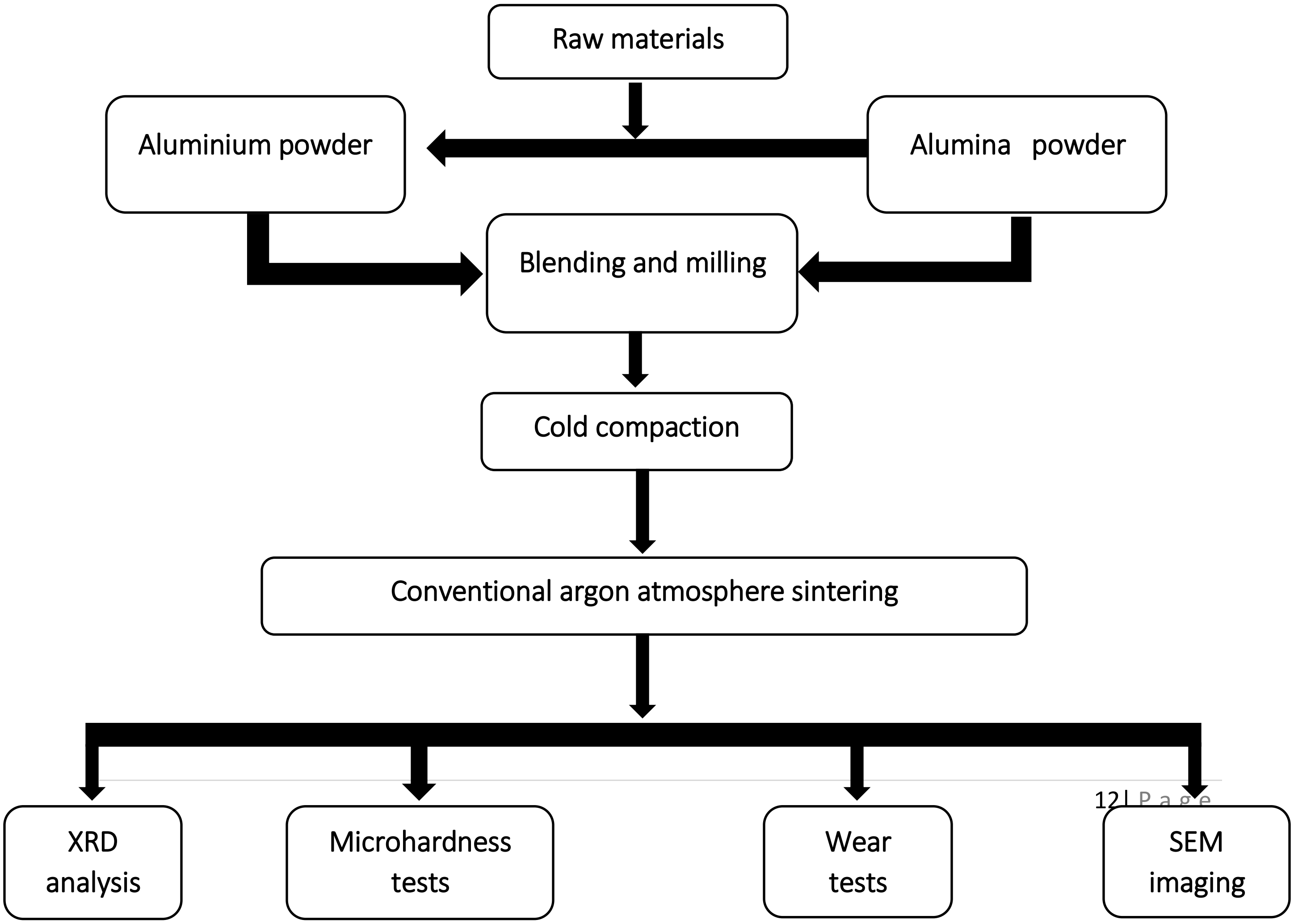
Here γ is a constant of order 1, G is the shear modulus, b is the Burger vector and ρ is the average dislocation density generated due to the CTE mismatch [23].

However, the strain may be relaxed by plastic straining at the matrix interface. This can lead to decohesion and cracking at the interface, particulate fracture, microvoid coalescence failure and shear fracture of matrix. All these lead to deterioration of properties and reduce reliability of the composite under critical applications [21-22].

3. Materials & Experimental procedure

3.1 Overview:

In this study, micro and nano alumina strengthened aluminium composites were prepared by conventional cold compaction and sintering route. This process involved, initially, proper weighing of the aluminium powder and corresponding amount of alumina powder equivalent to the required volume % in the composite. Thereafter, the respective amount of powders for each different composition separately were blended together, initially by mortar and pestle, followed by milling in the ball mill. Then, small amounts of each composition were taken and the green samples were prepared by uniaxial cold compaction, followed by sintering at a relatively high temperature. The resultant composite specimens were subjected to microhardness and wear tests. Thereafter, the samples were heated to a specified holding temperature, held for the required amount of time and finally quenched in different quenchants. This process was repeated for all compositions. Finally, the microhardness and wear tests were performed on all the quenched samples to compare differences with the unquenched samples and the microstructural studies were performed with both optical and electron microscopes.



3.2 Materials:

In the course of the experiments, the as received aluminium powder (LobaChemie purity > 99.7%, average size~22µm) along with alumina (Sigma–Aldrich, average size – 5.71 µm and <50 nm) powders were mixed and blended separately using agate mortar for 60 minutes to ensure homogeneous mixing. Alumina powders of both sizes were mixed with aluminium powder to prepare the micro and nano composite samples. In this process, aluminium powder containing 1, 3, 5, 7 vol. % of nano alumina (size < 50 nm) powder was mixed to prepare the nano composite powder. Similarly, aluminium powder was also mixed with 5, 10, 15, 20 vol. % of micro alumina (size < 5.71 µm) powder was to prepare the micro composite powder. The table below shows the different materials synthesized in the process –

Micro alumina reinforced composites	**Nano alumina reinforced composites**
Al + 5 vol. % Al_2O_3	**Al + 1 vol. % Al_2O_3**
Al + 10 vol. % Al_2O_3	**Al + 3 vol. % Al_2O_3**
Al + 15 vol. % Al_2O_3	**Al + 5 vol. % Al_2O_3**
Al + 20 vol. % Al_2O_3	**Al + 7 vol. % Al_2O_3**

3.3 Primary processing of the materials:

Here, the powders for each composition taken separately were initially blended together manually by an agate mortar and pestle. Thereafter, the powders were cold compacted to form the green pellets. These were then sintered in an argon atmosphere in a furnace to obtain the final composites.

3.3.1 Blending

Blending is an important step in powder metallurgy technique of preparing the composites to avoid segregation of reinforcements. This is more important for preparation of nano reinforced composites, because the nano alumina have a high tendency towards agglomeration due to their small size and resultant high surface energy. Blending was done manually, in an agate mortar and pestle by taking the required amount of aluminium powder and mixing small amounts of alumina powder, followed by proper blending until the alumina powder has been mixed properly. Additional amounts of alumina powder are added from time to time until the require amount of alumina has been added completely and the composite powder has uniform distribution through proper blending.

Thereafter, the manually blended powders were put into small enclosed plastic jars, containing 6 – 10 balls and blended in a turbula shaker mixer (T2F, LCR Hi Tester, Switzerland) for 8 hours at 45 rpm.

3.3.2 Cold compaction:

The homogenously mixed composite powders were then cold compacted in a uniaxial pressing machine (SoilLab) at a pressure of 400 MPa in a circular die set. The punch and die set were properly cleaned with acetone, followed by lubrication with powdered zinc stearate (dry), before adding the composite powder to the die and setting the punch. The punch die sets and loads were thereafter properly aligned and set along with the loads in the machine. Cylindrical test samples of 15 mm diameter were prepared with a holding time of 2 minutes. Thereafter, the samples were removed carefully taking care not to damage the fragile green samples.

[Fig-6]

3.3.3 Conventional furnace sintering:

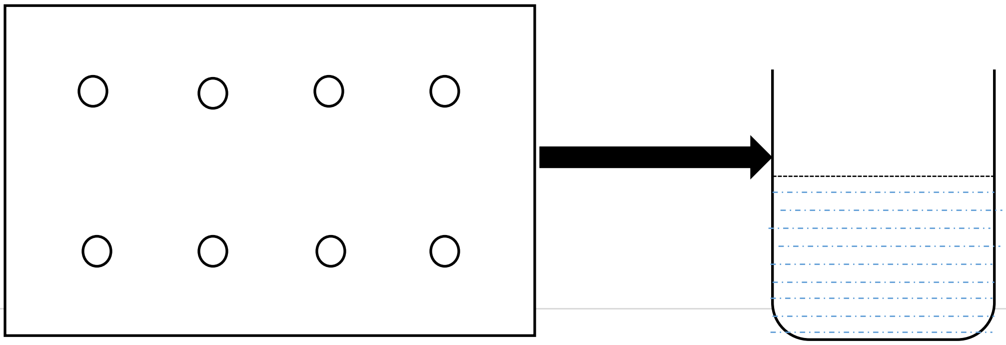
Sintering of the cylindrical pellets was carried out in a tubular furnace at a temperature of 550 ^{0}C (melting point of aluminium = 660 ^{0}C) for 60 minutes followed by cooling in the furnace at a rate of 5 ^{0}C/minute. The samples were loaded onto a plate or a crucible, prior to loading into the furnace. Slow cooling rate was adopted to avoid development of thermal stresses in the samples. Finally, the cooled samples were taken out of the furnace carefully.

[Fig-7]

3.4 Quenching:

The sintered cylindrical samples of each composition as specified in the tables were heated to the temperatures of 200 ^{0}C and 300 ^{0}C (for separate samples) at a heating rate of 6 ^{0}C/s, followed by a holding time of 1 hour at the specified holding temperature . Thereafter, the samples were carefully removed from the furnace and quenched in the specific quenching medium. Five quenching mediums were employed in this experiment which are –

- Air (natural convection)
- Brine (200 ml - 7 wt % NaCl)
- Liquid nitrogen
- Polymer quenchant (5 Vol. % Poly ethylene glycol in water)
- Oil quenchant (Servo engine oil)



[Fig-8]

3.5 Microstructural analysis:

In microstructural analysis, samples are prepared by metallographic techniques to observe the features with an electron microscope or an optical microscope.

3.5.1 Metallographic preparation:

For metallographic preparation of the samples, the samples were initially polished with emery paper starting from 1/0 (coarsest) grit size, followed by 2/0, 3/0 and finally 4/0 (finest) size. Polishing was done by polishing the sample son the paper repeatedly unidirectionally. When the next finer size paper was used, polishing was done in a direction perpendicular to the previous polishing direction. This was repeated until complete polishing of the samples. After the paper polishing, the samples were subjected to cloth polishing with Brasso liquid. Final cloth polishing is done with a diamond spray liquid that ensures a fine mirror like finish to the surface of the samples. The final prepared sample has a bright mirror like finish on the polished surface.

3.5.2 X ray diffraction analysis:

X-ray diffraction was carried out, on the sintered composite samples, using the diffractometer (PANalytical model: DY-1656). CuK$_{\alpha}$ (λ=1.542A°) radiations were used and the diffraction was carried out with a scan speed of 2°/minute.

3.5.3 Scanning electron microscopy:

SEM JEOL 64480LV Scanning Electron Microscope (SEM), was used to investigate the microstructure of the quenched samples. Using an accelerating voltage of 15 kV and SE imaging mode, various positions of the samples were checked for the alumina distribution and changes due to quenching in the samples. Images were captured at different magnifications.

3.6 Mechanical characterization:

Various microhardness test were performed on the samples to determine the changes due to quenching.

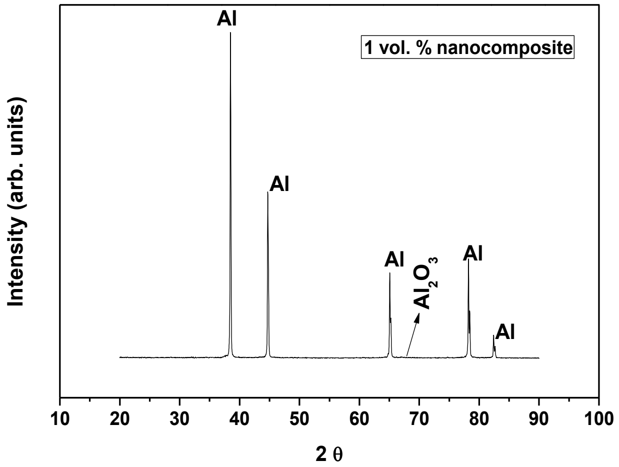
3.6.1 Microhardness testing:

The micro-hardness values of every specimen was determined by Vickers hardness tester (Leco LV 700). For this purpose, four readings were taken at four different points on the specimen and average and standard deviation were calculated. The readings were taken, both before and after the quenching experiments, to determine changes in mechanical properties. A load of 25 gf and a dwell time of 5 seconds were used and total hardness was obtained by measuring the size of the indent in Vickers hardness (HV).

4. Results and discussion

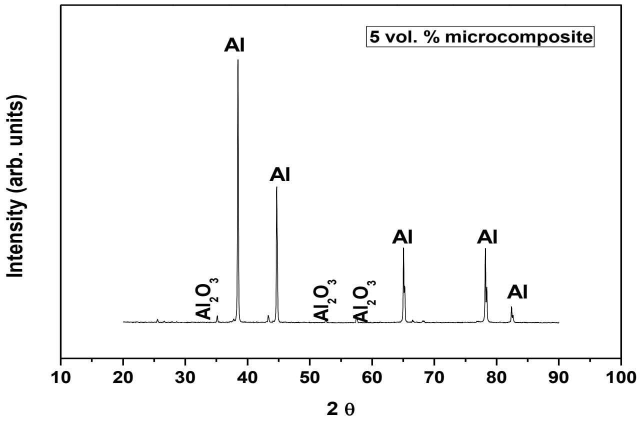
4.1 XRD analysis:

The XRD analysis was performed to know about the phases present and possible reaction products.



[Fig-9(a)]

Intensity peaks in the XRD plot indicate presence of only Al_2O_3 and Al as the products in the samples. This means no side reaction took place and concentrations of other impurities are negligibly small in the composite. Due to the low volume fraction of the Al_2O_3, the peaks of alumina are very small compared to Al peaks in the composite.



[Fig-9(b)]

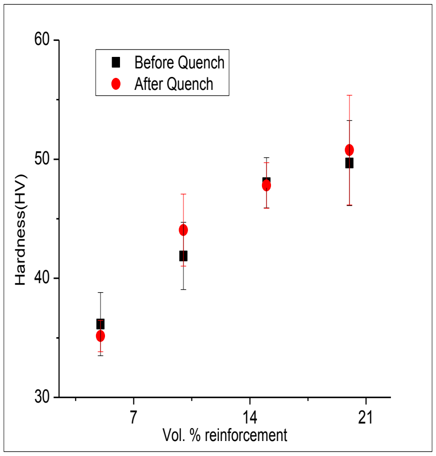
Like the previous case of nanocomposites, the micro reinforced composites also show only the presence of aluminium and alumina in the samples. Hence, the role of other impurities in affecting the properties of aluminium composite can be ruled out by the XRD analysis. Also due to the slightly higher concentration of alumina in this case (5%), the Al_2O_3 peaks are slightly higher in the XRD plots.

4.2 Hardness:

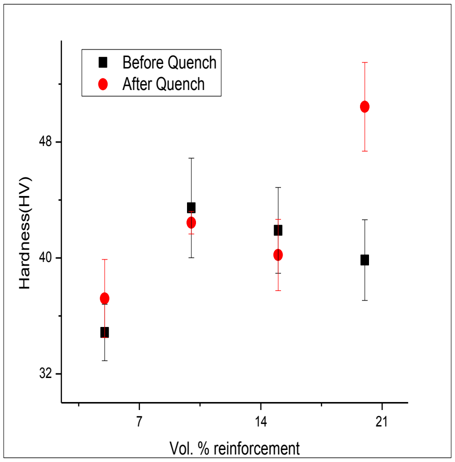
4.2.1 Air quenched samples-

Micro alumina reinforced samples –

The plots of hardness vs the compositions for the air quenched samples are –



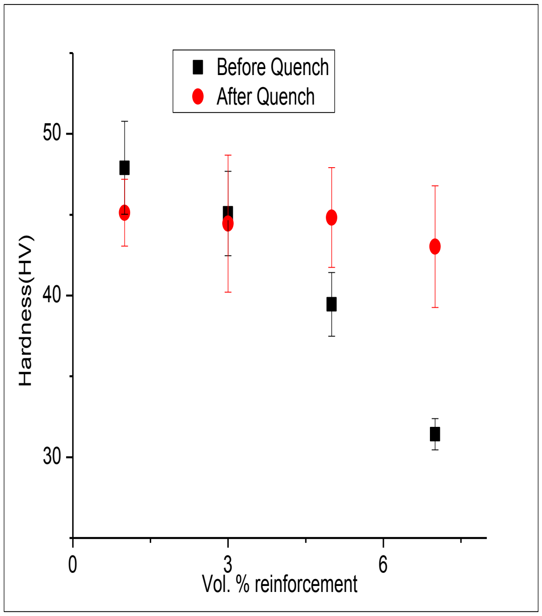
[Fig-10(a)]



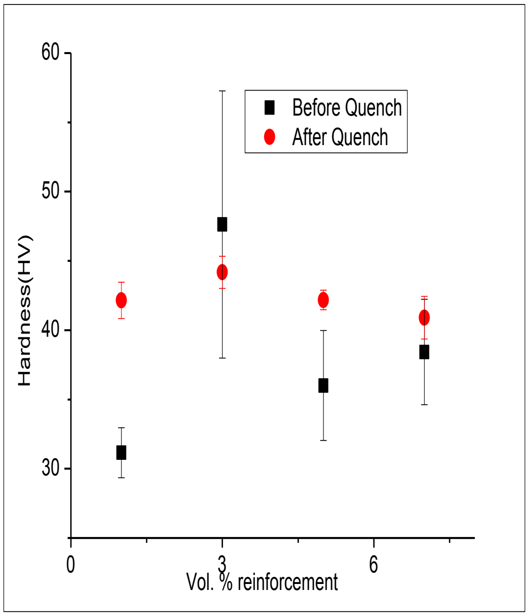
[Fig-10(b)]

As can be seen from the plots, the variation in hardness for quenching from the 200 ^{0}C holding temperature is minimal. Apart from the 15 vol. % sample, rest of the others do not show much change in hardness, indicating lack of thermal shock for air quenching at 200 ^{0}C. A similar phenomenon occurs with the 300 ^{0}C holding temperature samples, except for the 20 vol. % alumina samples, that show considerable increase in hardness (39 to 50) after quench. This may be due to the larger number of dislocations generated on account of the greater particle matrix interface. Hence, increasing the reinforcements, we can expect a greater increase in hardness due to larger number of dislocations generated at the particle matrix interface. But, the lack of such behaviour in 10 and 15 vol. % samples may be interpreted as lack of good bonding between the reinforcement and matrix, due to which dislocations are not generated during quenching.

<u>Nano alumina reinforced samples –</u>



[Fig-11(a) 200 ^{0}C holding temperature

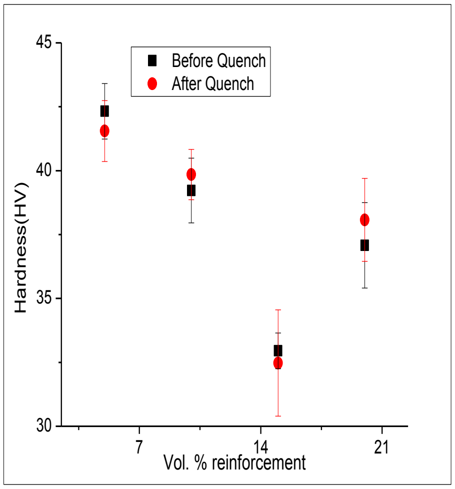


[Fig-11(b)] 300 ^{0}C holding temperature

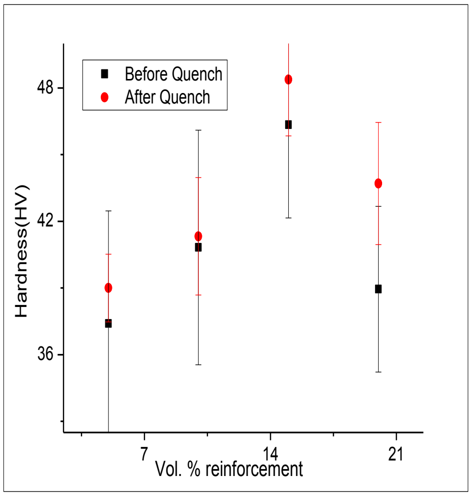
As can be observed from the above graphs, there is a clear trend towards higher hardness in nano samples after quenching. However some anomalous softening is still observed in 1 or 2 samples. This may be due to particle agglomeration or decohesion. But as has been stated beforehand, there is clearly a greater increase in hardness for higher vol. % nano samples quenched from 200 ^{0}C which is due to the greater interfacial area. But quenching from 300 ^{0}C hardness increase is lower for higher vol. % which may be due to some damage at matrix particle interface. However, due to the slow cooling rate in air, such observations may not represent significant differences.

4.2.2 Oil quenched samples-

Micro alumina reinforced samples –



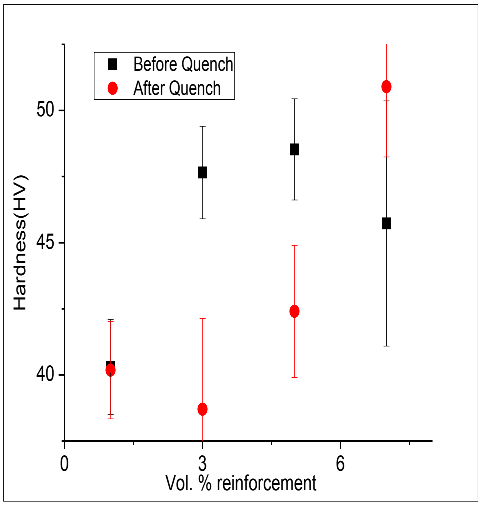
[Fig-12(a)] 200 ^{0}C holding temperature



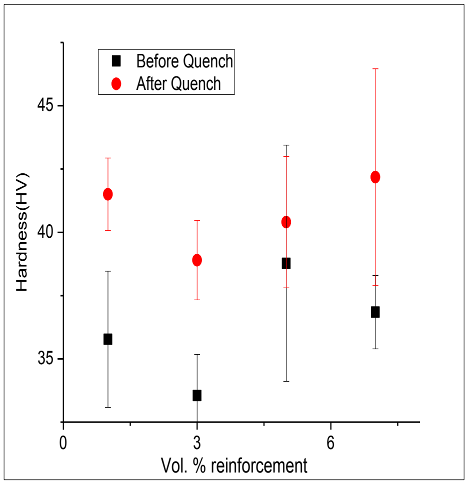
[Fig-12(b)] 300 ^{0}C holding temperature

As can be observed, variations in hardness do not seem to follow any well-defined trend for a holding temperature of 200 ^{0}C, while, there is a clear increase of hardness for a holding temperature of 300 ^{0}C. This indicates that due to the higher ΔT in this case, there is a greater nos. of dislocation generation, leading to increased hardness. Also relative increase in hardness is higher for higher vol. % reinforced samples, likely due to greater interfacial area, leading to increased nos. of dislocations.

Nano alumina reinforced samples –



[Fig-13(a)200 ^{0}C holding temperature

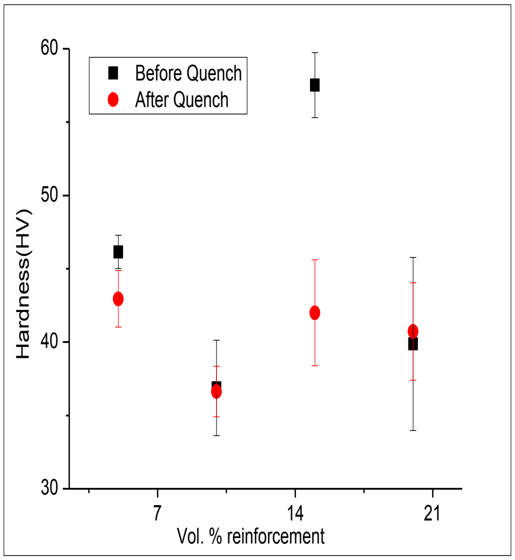


[Fig-13(b)] 300 ^{0}C holding temperature

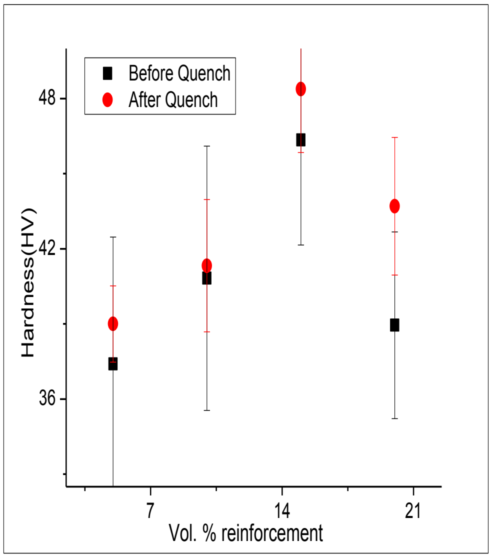
As can be seen, for 200 ^{0}C holding temperatures, the softening in some samples can be interpreted as either the effect of agglomeration or due to decohesion or damage at the particle matrix interface. However, there is an increase in hardness in all cases for 300 ^{0}C holding temperatures, indicating greater number of dislocations after quenching. Also the highest relative change in hardness is for the higher vol. % samples, keeping in line with the trends observed for the previous cases.

4.2.3 Polymer (5 vol. % Poly ethylene glycol) quenching –

Micro alumina reinforced samples –



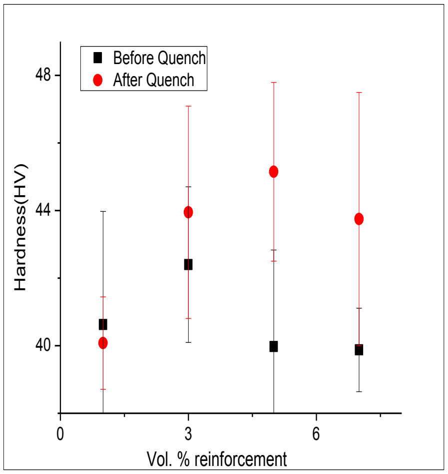
[Fig-14(a)] 200 ^{0}C holding temperature



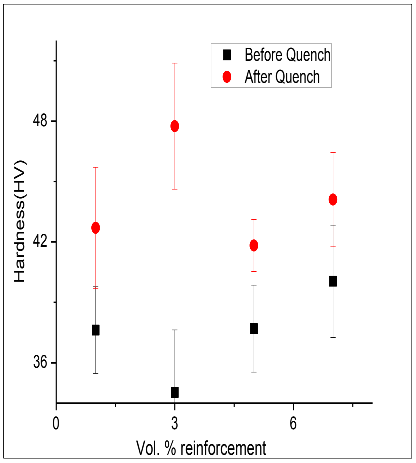
[Fig-14(b)] 300 ^{0}C holding temperature

Polymer quenched samples from 200 ^{0}C show an irregular trend, indicating lack of hardening due to dislocation generation. However, quenching from 300 ^{0}C shows an increase in hardness for all cases, with a higher increase in hardness for higher vol. % samples like the previous cases.

<u>Nano alumina reinforced samples –</u>



[Fig-15(a) 200 ^{0}C holding temperature

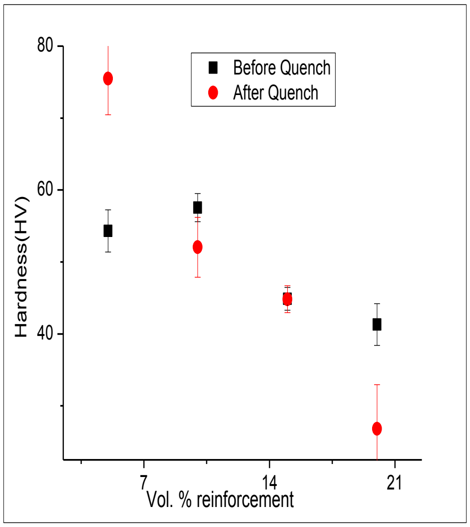


[Fig-15(b)] 300 ^{0}C holding temperature

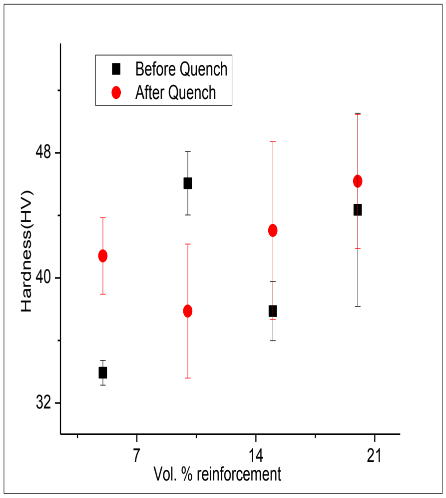
Here also, there is an increase in hardness for all the vol. % reinforcement cases for samples quenched from 300 ^{0}C, while there is an increase in 3 cases for quenching from 200 ^{0}C. However, there is no proper trend of greater hardness change for higher vol. % reinforcement for quenching from 300 ^{0}C.

4.2.4 Brine (200 ml – 7 wt % NaCl solution) quenching –

<u>Micro alumina reinforced samples –</u>



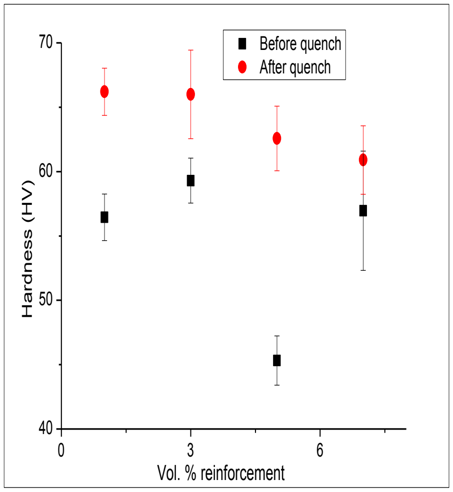
[Fig-16(a)] 200 ^{0}C holding temperatur



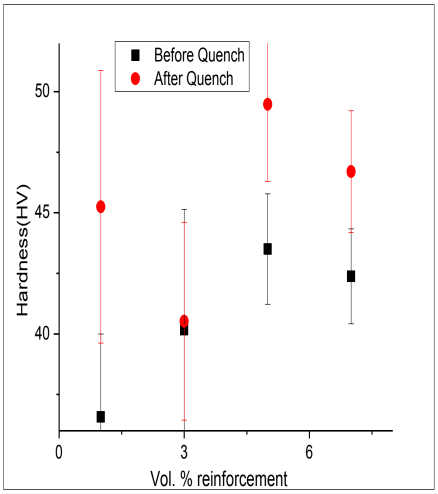
[Fig-16(b) 300 ^{0}C holding temperature

As can be observed from the above graphs, 300 ^{0}C holding temperature is more effective in inducing higher hardness in the samples compared to 200 ^{0}C holding temperature.

<u>Nano alumina reinforced samples –</u>



[Fig-17(a)200 ^{0}C holding temperature

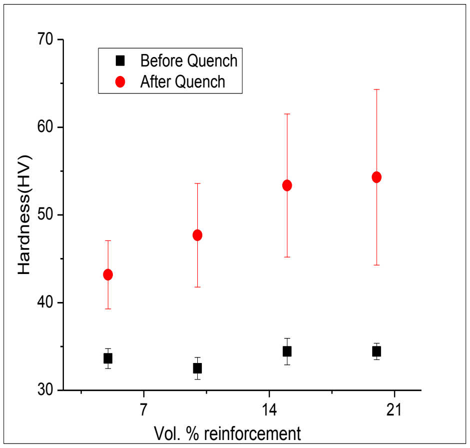


[Fig-17(b)] 300 ^{0}C holding temperature

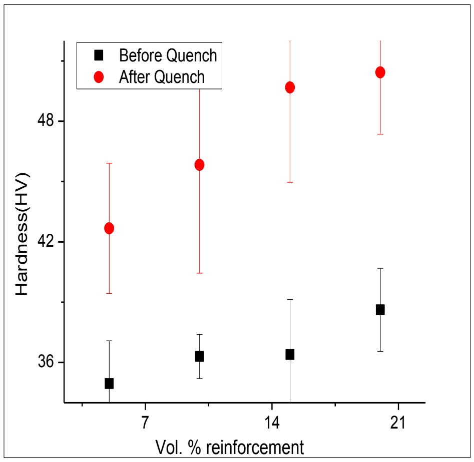
As is observed in this case, in all cases, here there is a considerable increase in hardness compared to some of the previous quenchants, where there was a negligible change or softening. This is due to the higher quenching rate by brine which provides lesser time for strain relaxation, thereby increasing the dislocation density.

4.2.5 Liq. N_2 quenching:

<u>Micro alumina reinforced samples –</u>



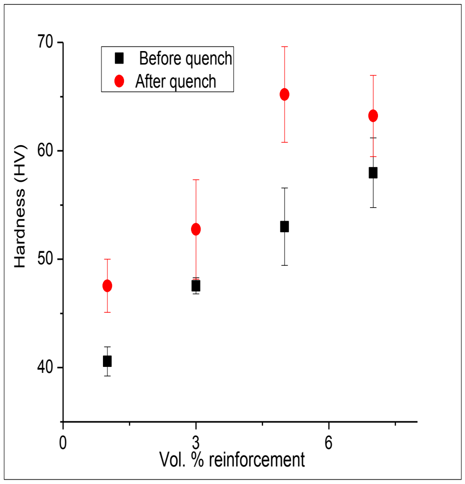
[Fig-18(a)] 200 ^{0}C holding temperature



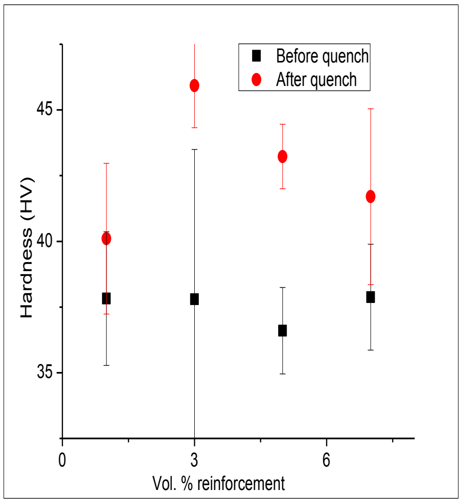
[Fig-18(b) 300 ^{0}C holding temperature

A clear trend emerges in this case of higher quenched hardness and higher increase in hardness with increase in vol. % of reinforcement for both holding temperatures at 200 ^{0}C and 300 ^{0}C. This indicates increased dislocation density as well as the role of interfacial area in determining the hardness increase.

Nano alumina reinforced samples –



[Fig-19(a)]200 ^{0}C holding temperature



[Fig-19(b)] 300 ^{0}C holding temperature

Like for the micro samples, nano composite samples quenched in liq. N_2 also show increasing hardness and a greater increase in hardness for higher volume % of reinforcement. The slightly lower increase in hardness for the 20 vol. % composites compared to 15 and 10 vol. % composites for both 200 ^{0}C and 300 ^{0}C may be due to some decohesion or agglomeration of the particles.

The above observations clearly point out that, with increase in quenching rate, the hardness increases and so does the increase in hardness for higher vol. % reinforcement samples. In some cases, the random softening after quenching can be due to decohesion of the particles from the matrix, before thermal misfit dislocations are generated, thereby resulting in the matrix behaving as an unreinforced material.

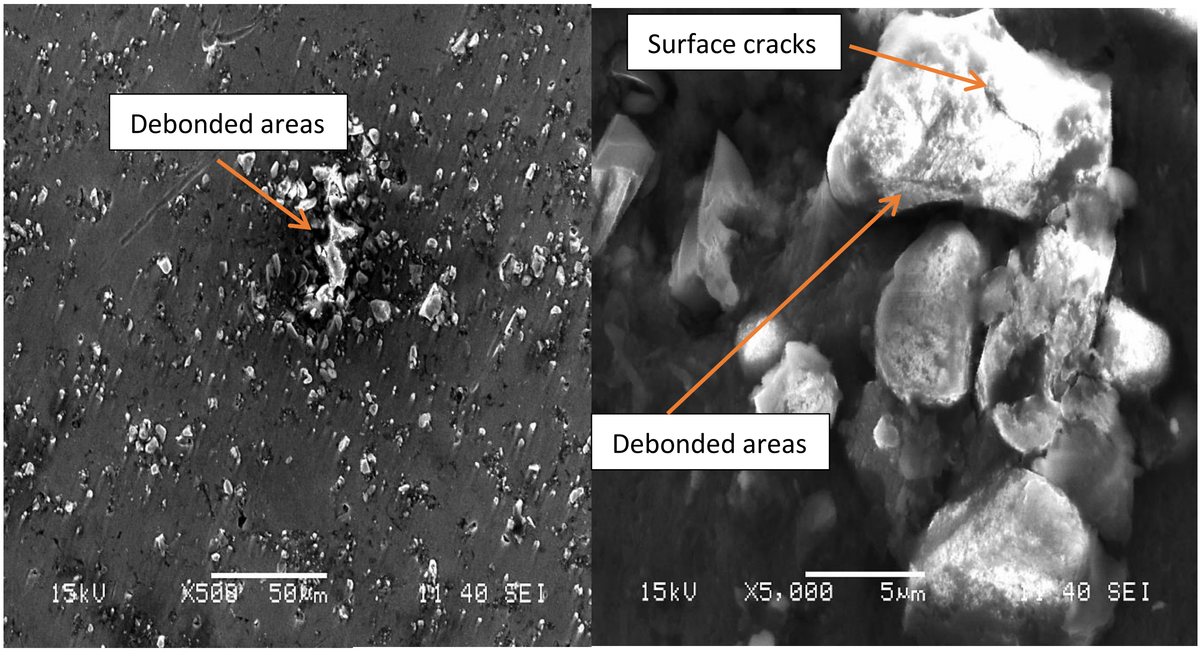
Solid solution strengthening as a cause of hardness seems unlikely because no alloying elements or impurities were added during the process. Also natural ageing or other reactions at interface are unlikely because of the unreactive nature of the reinforcement with the matrix. The change in hardness depends on the following factors –

- Dislocation generation due to thermal mismatch.
- Interfacial damage due to different thermal expansion.
- Previous state of strain hardening of the sample prior to quenching.

The thermal mismatch dislocations increase the hardness, while interfacial damage and decohesion or fracture of the reinforcements are likely to reduce the hardness due to reduced ability of the reinforcement particle to resist matrix deformation. The third factor, may lead to an increase in hardness for a composite with little prior work hardening or a decrease in hardness for a heavily work hardened composite. The back stress provided by the particles plays an important role in this case. The first two factors increase with an increase in volume fraction of reinforcement or interfacial area (higher for nano reinforcements).However, the overall back stress in the matrix is higher for the higher vol. fraction alumina composite than for a lower volume fraction alumina composite. Hence, depending on a balance between all these factors, the composite may exhibit an increase in hardness (mostly) or a decrease in hardness (few cases).

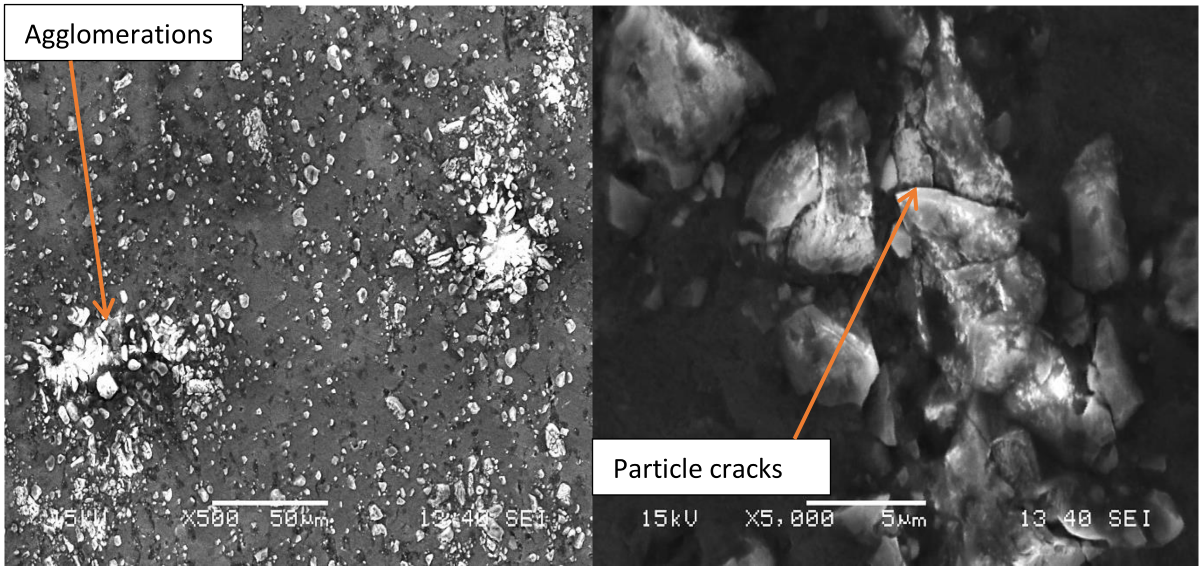
4.3 Microstructure:

<u>Micro alumina reinforced samples –</u>



5 M samples post quenching images

[Fig-20(a)] [Fig-20(b)]

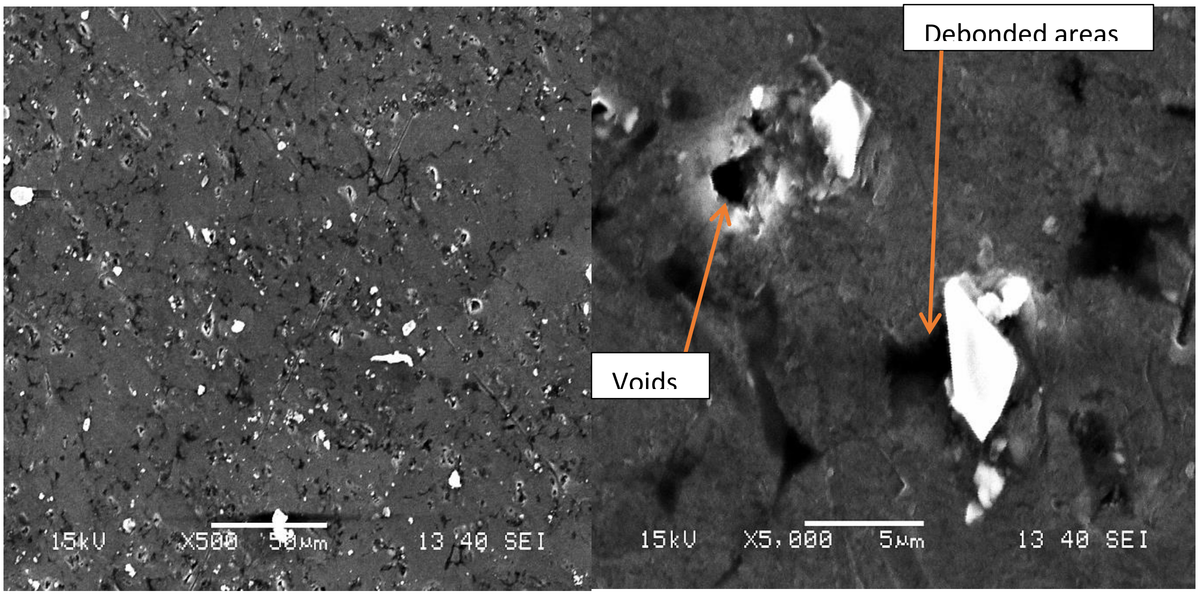


20 M samples post quenching images

[Fig-21(a)] [Fig-21(b)]

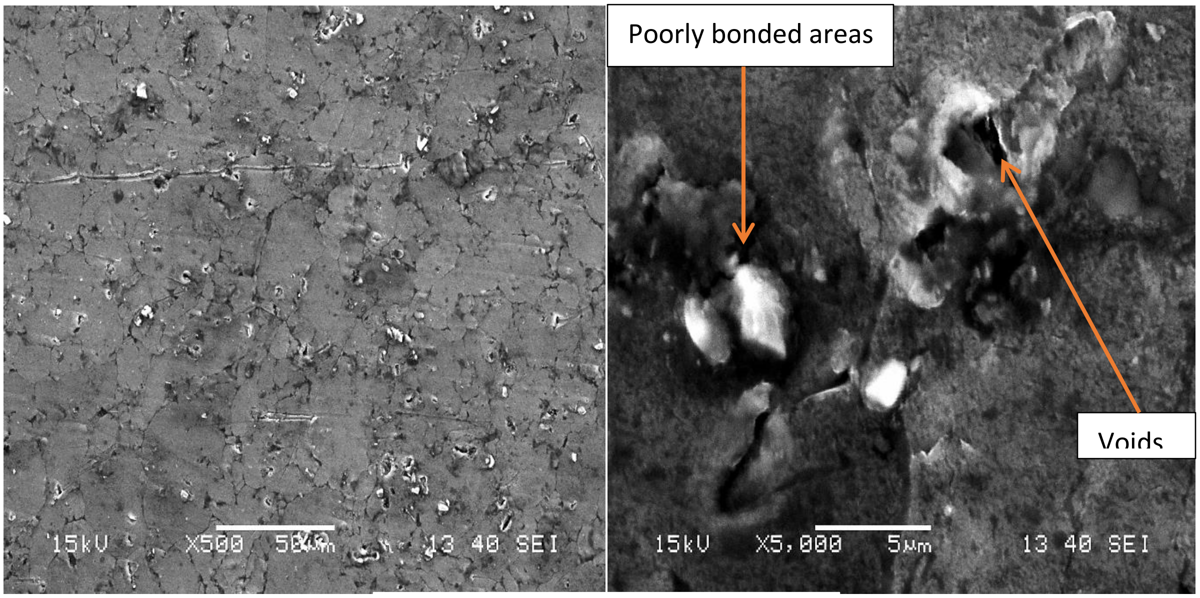
The above images for Liq N_2 quenched samples held at 200 ^{0}C show some agglomeration in the samples. However the 5000 X images clearly show debonding and decohesion along the interface as represented by the dark areas. Some cracks are visible in the reinforcements, for the 20 M samples, likely the result of non-uniform contraction during quenching. Both of these factors are likely to reduce effectiveness of the reinforcements in maintaining matrix hardness.

<u>Nano alumina reinforced samples –</u>



1N samples post quenching images

[Fig-22(a)] [Fig-22(b)]



3N samples post quenching images

[Fig-23(a)] [Fig-23(b)]

The above images show a relatively uniform distribution of the nano particles in the matrix. However, the high magnification images show clear debonding at the interfaces. Non uniform deformation areas near the particle matrix interfaces are also visible in the high magnification 3N images. All these indicate, inspite of matrix hardening, interfacial damage is likely to reduce effectiveness of reinforcement in improving the strength.

5. Conclusion

1. Increase in hardness is observed in most cases of quenching for the aluminium-alumina composites. This hardness increase is likely a result of thermal misfit dislocations generated at the interfaces, and hence the increase shows a greater increase for higher vol. % samples.
2. In a few cases, the quenched hardness is lower than the pre quenched hardness. The likely reason is interfacial damage during quenching which reduces effectiveness of the reinforcements in resisting matrix deformation.
3. Hardness increases with increase in quenching rate, showing maximum relative increase for liquid nitrogen quenching from 300 ^{0}C holding temperature.
4. Wear –
5. Microstructural characterisations show debonding and decohesion at the interfaces and quench cracks in the structure.

6. Recommendations for further work

1. Effect of quenching in age hardenable aluminium composites is a key area of further investigation. The role of thermal dislocations in nucleating precipitates and improving ageing can be an interesting area for further research.
2. Residual stress analysis in the quenched samples and their effects on fatigue properties as well as impact properties can be analysed and solutions can be suggested to improve the reliability of such composites for critical applications.
3. Mathematical modelling and simulation of residual stresses and mechanical properties of quenched composites can be done to predict likelihood of failure of such composites.
4. Quench properties of composites synthesized by in situ techniques relative to other techniques can be investigated and compared to observe the effects of stronger bonding in the in situ composites.

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