

University Of Jordan  
Faculty Of Graduate Studies

*w/w*

# The Effect Of Some Oxides Additions On The Mechanical Properties of Aluminum

BY

Firas Jaber

SUPERVISOR

Prof. Adnan Kilani

Submitted in partial fulfillment of the requirements for the degree  
of Master of Science in Mechanical Engineering.

Faculty of Graduate Studies,

University of Jordan

Amman Jordan

May, 1994

*w/w/c*

# Committee Decision

This thesis was defended successfully on 18th May, 1994.

## COMMITTEE MEMBERS

## SIGNATURE

1. Dr. Adnan I. O. Zaid Kilani (supervisor)

Professor

Industrial Engineering Department

University of Jordan.

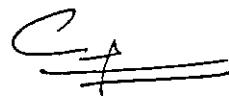


2. Dr. Saad Moh'd Habali

Associate Professor

Mechanical Engineering Department

University of Jordan.



3. Dr. Salah Elddin Alsayed

Associate Professor

Vice Dean of the Engineering Faculty

Applied Science University



# Acknowledgement

With deep pleasure I express my gratitude to those who helped me during the preparation of this work. Special thanks are dedicated to my supervisor Prof. Adnan Kilani, who without his support, encouragement, and advice, my work could have been more difficult. Thanks are due to Dr. Ahmad Abed Raouf, Eng. N. S. AL-Hajeid and Mr Mohamad Arayda. Finally, I should not forget to express my gratitude for the support given to me by all my friends throughout this work.

# Contents

Committee Decision . . . . .	ii
Acknowledgement . . . . .	iii
Table of contents . . . . .	v
List of figures . . . . .	vii
Nomenclature . . . . .	x
Abstract . . . . .	xi
1 Introduction . . . . .	1
2 Literature Review . . . . .	3
2.1 Coating and Reactive Agents . . . . .	3
2.2 Vortex and Compocasting Techniques . . . . .	5
2.3 Pressure Infiltration . . . . .	8
2.4 Significance of The study . . . . .	16
3 Theoretical Considerations . . . . .	17
3.1 Wetting . . . . .	18
3.2 Distribution and Uniformity . . . . .	21
3.3 Solidifying Techniques . . . . .	23
4 Materials, Equipment and Experimental Procedure . . . . .	28
4.1 Materials . . . . .	28
4.2 Equipment . . . . .	28
4.3 Experimental Procedure . . . . .	30

		v
4.3.1	Wetting Optimization . . . . .	30
4.3.2	Fabrication of Al/Al <sub>2</sub> O <sub>3</sub> Metal Matrix Particulate Reinforced Composite . . . . .	30
4.4	Metallurgical Examination . . . . .	35
4.5	Mechanical Behaviour Tests . . . . .	36
4.5.1	Wear Analysis . . . . .	37
4.5.2	Mass Loss with Time . . . . .	37
5	Results and Discussion . . . . .	40
5.1	Wetting optimization . . . . .	40
5.2	Rate of Addition . . . . .	43
5.3	Mixing Speed and Angle of the Impeller . . . . .	43
5.4	Solidifying Techniques . . . . .	47
5.4.1	Injection Mixing . . . . .	47
5.4.2	Centrifugal Casting Technique . . . . .	50
5.4.3	Vortex Technique . . . . .	53
5.4.4	Rheocasting Technique . . . . .	57
5.4.5	The Modified Vortex Technique . . . . .	59
5.4.6	Modified Vortex Technique With Grain Refiner . . . . .	61
5.5	Mechanical Behaviour And Hardness Test . . . . .	65
5.6	Machinability . . . . .	65
5.7	Wear Analysis . . . . .	67
5.7.1	Wear Rate Vs. Spring load and Wear rate Vs. Slid- ing Speed . . . . .	67
6	Conclusions and Recommendations for Future Work . . . . .	85
6.1	Conclusions . . . . .	85
6.2	Recommendations . . . . .	87
	REFERENCES . . . . .	88
	Abstract(Arabic) . . . . .	93

5.12 Compocast Technique Results, X95 . . . . .	58
5.13 Modified Vortex Technique Results . . . . .	62
5.14 Modified Vortex Technique with Refining Agent, Top Section, X25 . . . . .	63
5.15 Modified Vortex Technique with Refining Agent, Bottom Section, X25 . . . . .	63
5.16 Modified Vortex Technique with Refining Agent, Longitudinal Section, X100 . . . . .	64
5.17 Electroscanning photo for $Al_2O_3$ particle . . . . .	64
5.18 True Stress True Strain curve . . . . .	69
5.19 Ra Vs. Feed Rate . . . . .	70
5.20 Ra Vs. Depth of Cut . . . . .	71
5.21 Ra Vs. Sliding speed . . . . .	72
5.22 Mass Loss with Time . . . . .	73
5.23 Mass Loss with Time . . . . .	74
5.24 Mass Loss with Time . . . . .	75
5.25 Mass Loss with Time . . . . .	76
5.26 Mass Loss with Time . . . . .	77
5.27 Mass Loss with Time . . . . .	78
5.28 Mass Loss with Time . . . . .	79
5.29 Wear Rate with load . . . . .	80
5.30 Wear Rate with sliding speed . . . . .	81
5.31 roughness with Load and Sliding speed . . . . .	82
5.32 Speed calibration chart . . . . .	83
5.33 Load calibration chart . . . . .	84

# Nomenclature

$V_f$	Volume fraction [%]
$W$	Wear Rate [mg/hour]
$R_a$	Average Microroughness [ $\mu m$ ]
$D$	Depth of Cut [mm]
$V_s$	Sliding velocity [mm/s]
$f$	Feed rate [mm/sec]
$MMC$	Metal Matrix Composite
$d$	Impeller dia. [mm]
$D_v$	Vortex dia. [mm]
$D_c$	Crucible dia. [mm]
$d_i$	Impeller depth. [mm]

- Modified technique.

Comparison between the results of these different techniques is held with respect to the volume fraction, particle distribution, soundness and finally microsegregation.

It was found that the use of refining agent (Titanium) with the range 0.05 % to 0.1 % has optimized the particle distribution by lowering the microsegregation effect to negligible levels.

Mechanical behaviour and hardness tests are performed on both composite and aluminum matrix, and the results showed an improvement in both hardness and strength.

Surface roughness measurements were taken on turned composite and pure aluminum specimens with variable cutting parameters such as feed rate, depth of cut and sliding speed, from which it was found that the Alumina particles caused enhancement to the surface roughness.

It was found that increasing the feed rate or the cutting depth resulted in increased surface roughness, whereas increasing the sliding speed improved the resulting surface roughness, till a critical speed value is reached (4.5  $m/s$ ), after which no further improvement in the surface roughness is achieved.

Finally wear tests were conducted on the composite specimens with load range 2.5  $N$  to 15  $N$  and sliding velocity range of 1.57  $m/s$  to 3.93  $m/s$  . A stable load range was found and critical velocity is determined (2.36  $m/s$ ) below which the wear rate decreases by increasing the sliding speed. .



# Chapter 1

## Introduction

The increased demand on materials with several and superior properties to withstand the different and combined loading conditions in addition to environmental ones that exist in specific applications of highly sophisticated industries become an essential requirements nowadays.

The incorporation of two (or more) different materials to produce a new one having superior properties in addition to its constituents properties is called a composite material. If one of these constituents is a metal then the composite material is called a Metal Matrix Composite (MMC). therefore the addition of ceramic particles (oxides or carbides) to a molten aluminum is regarded to fall within the field of MMCS [1,2].

Systematic design and synthesis procedures were developed to achieve a unique combination of engineering properties such as high temperature strengths, fatigue strength, damping properties, electrical conductivity, thermal conductivity and coefficient of thermal expansion[1].

Aluminum materials are preferred structural materials for their comparably high strength-weight ratio. The use of these materials is particularly important in transportation -street vehicles, rail vehicles, air planes - in order to reduce weight and energy consumption.

Additionally significant improvements in wear resistance, coefficient of friction , and hardness (over a range of temperature) of aluminum

alloys can be achieved through synthesis of these alloys with a variety of particles like graphite, mica, talc, as well as hard ceramic materials such as zircon, alumina, silicon carbide [3].

The resulting properties make these composites suitable for high performance aero-space and defense applications. It has been reported that the addition of 1 % alumina ( 7 um particles size) to an aluminium alloy can lead to increased hardness and four- fold increase in strength at 350 C° [4]. Thus the motivation to investigate the effective technique of adding some of these ceramic particles to a molten aluminum is now clearly justified.

RECORD COPY	ACC. NO. 436768-65	UNIVERSITY OF JORDAN LIBRARY	ORDER NO. 3121
	DEALER مركز الكتب		DATE
	PRICE	AUTHOR	
		TITLE & ED.	
	PLACE	PUBLISHER	
	DATE	NO. OF COPIES	
	RECEIVED : INVOICE NO. & DATE		14 JUN 1994

# Chapter 2

## Literature Review

Until the beginning of the eighties aluminum-ceramic particles composites were generally made by conventional powder metallurgy methods, since then attempts have been made to prepare these composites by liquid metallurgy.

As it will be clarified in Chapter 3, there were difficulties in optimizing the wetting, bonding, and distribution during the fibre or particle incorporation.

### 2.1 Coating and Reactive Agents

436763

Several approaches have been employed to improve the poor wetting between liquid aluminum and ceramic particles. However, compatibility and bonding between the fiber or particle and the metal matrix in these systems are induced by the chemical vapor deposition of a thin layer of titanium and boron onto the fibers or particles to achieve wetting. These coated fibers or particles are air unstable because of rapid oxidation of the titanium boron coating when exposed to air and because molten metal does not wet the fibre or particle. To overcome this difficulty, air stable coatings of SiO<sub>2</sub> that are wetted by Mg have been used. Titanium-boron coatings have also been used for graphite/aluminum, Al<sub>2</sub>O<sub>3</sub>/aluminum, and Al<sub>2</sub>O<sub>3</sub>/lead metal matrix composites [5].

Continuous adherent metallic coatings (for example copper and nickel) on several non wetting particles such as graphite, shell char and mica, were found to improve the melt-particle wettability and allow high percentages of these particles to be recovered in solidified castings [6,7].

Badia [8] used nickel coating on the alumina particles to improve wetting between ceramic particles and liquid aluminum, whereas Mcevoy et al. [9] have reported that MgO displays good wettability for liquid aluminum, and so attempts have been made [10] to obtain a thin coating of MgO layer around the  $Al_2O_3$  particles before dispersion, so that a good retention of  $Al_2O_3$  particles in the composite could result. This objective was achieved through mechanical mixing of coarse  $Al_2O_3$  particles (8-80  $\mu m$ ) with submicron MgO powder (0.3  $\mu m$  to 0.4  $\mu m$ ) for a period of 30/35 minutes in a blender. The MgO coating so obtained was found to be one to two layers thick. The two powders  $Al_2O_3$  and MgO was taken in definite proportions before mixing to optimize the powder fractions for maximum  $Al_2O_3$  recovery.

Alumina-reinforced aluminum, copper, lead, and zinc composites as well as several particle-filled MMcs have been synthesized by using reactive agents [11,12].

Several workers [11-16], have used Mg as the reactive agent for the aluminum alumina composite system, Mg forms the spinel  $[MgO, Al_2O_3]$ . Similarly, lithium was used [17-20] to form the spinel  $[LiO, 5Al_2O_3]$ , since lithium can rapidly stimulates such a suitable reaction without requiring oxygen introduction by agitation. Again the incorporation of lithium can be associated with certain fabrication and handling problems.

Recent study [21] shows that Mn displays an improving wettability characteristics.

## 2.2 Vortex and Compocasting Techniques

Surappa et al. [4] have developed a casting technique for preparing Al-illte and Al-SiC particle composites. The method consists of stirring uncoated but suitably heat treated ceramic particles of varying sizes from (10  $\mu\text{m}$ -200  $\mu\text{m}$ ) in molten aluminium alloys (Vortex Technique), followed by casting of the composite melt. and microscopic distribution of the various ceramic particles have been reported. Wear tests have been conducted, hardness was increased for these composites, while the tensile strength was decreased due to the addition of 3 %  $\text{Al}_2\text{O}_3$ . Adhesive wear rates of the Al-alloys has decreased due to the addition of  $\text{Al}_2\text{O}_3$  particles.

Ghosh et al.[14] have investigated the effect of the size and volume fraction, Vf, of alumina particles and porosity on the tensile strength of Al-4% Mg- alumina compocast particulate composite.

The contribution of porosity to the reduction in strength of the composite at various levels of alumina content was observed. For a composite containing a lower level ( less than 7% vol. ) of alumina particles a rapid decrease in the value of tensile strength was observed, however with increase of Vf of alumina, the rate of decrease in tensile strength slows down.

Ghosh et al. [13] have studied the influence of mixing parameters such as holding temperature, stirring speed, size of the impeller and the position of the impeller inside the melt on the microstructure at different regions of Al (Mg)  $\text{Al}_2\text{O}_3$  compocast ingots, at all the combination of mixing parameters there always exists a tendency to form dendritic microstructure especially at the top of the ingot. The extent of dendritic growth at the top of the ingot increases with reduction in holding temperature, stirring speed, and size of the impeller and the placement

of the impeller close to the surface of the slurry. The regions of large primary solid particles and coarse dendrites observed at lower holding temperature create inhomogeneity in the distribution of alumina particles by restricting the entry of incorporated alumina particles in these zones.

Lee and Kim [22] have processed the Al- $Al_2O_3$  metal matrix composite by compocasting technique with a fibre volume fraction ranging from 10-20 %. The alumina whiskers used were added to a vigorously agitated molten matrix alloy. Mechanical tests performed at room temperature show that the modulus of elasticity and the ultimate tensile strength of the composite at 20 % volume fraction were improved about 41 % and 38 % respectively as compared to the un-reinforced materials. Ductility and impact resistance were lowered, the fibers were retained in the matrix materials with good distributions. Lack of preferred orientation of fibers and porosities were observed in conventional compocasting. This situation might be improved by rolling or extrusion.

Ghosh et al.[16] have prepared the Al (Mg)  $Al_2O_3$  particles composite by the compocasting technique carried out under various holding temperature and stirring speeds. During preparation of the composite a reacted layer has been found to form at the particle matrix interface, the thickness of the reacted layer has been found to vary with a change in holding temperature and stirring speed. The mechanism of formation of the reacted layer on the incorporated alumina particles, primarily composed of  $MgAl_2O_4$ , and identified by x-ray diffraction technique is explained, it has been observed that the extent of the reacted layer at the particle/Matrix interface decreases with an increase in the stirring speed and an increase in the holding temperature due to mechanical erosion caused by particle collisions in the turbulent melt.

Kiuchi et al.[23] in their research have investigated the complex processing of metal powder and ceramics particles based on mashy-state by forging and hot and/or cold rolling in order to develop a new manufacturing technology of particle reinforced metal with high deformability. In the process, mashy-state forging is adopted in order to get preformed composite billets. Then the billets are subjected to hot and/or cold rolling and made into composite sheets. Aluminum alloys (6061, 2014) are used as the matrix and aluminum oxide ( particle size is 5-149  $\mu\text{m}$  ) is used as reinforcing particle. The composite sheets were investigated regarding their internal structure, hardness and formability each as deformability in a bending test. Mashy-state forging is effective in getting good bonding between matrix and reinforcing particles. The high reduction in thickness given to the billets by rolling is necessary to improve formability.

Yang et al. [11] have incorporated amounts of 1-20 % bauxite particles into Al-12Si-1.4Cu-1.3Mg alloy by the rheocasting method. Bauxite is a starting raw material of alumina with excellent chemical and thermal stability at high temperature, a low coefficient of thermal expansion and high hardness . The abrasive wear resistance increased with increasing the amount of particulate addition. Under low and moderate loads, the wear resistance of the composite containing 20% wt. was comparable with that of carbon steel. However under high loads, carbon steel was superior to the composites in wear resistance. Particle clustering was observed in the composites owing to the small size of the bauxite particles; it had little influence on the wear resistance and mechanical strength of the composite. The sliding wear of composite containing both bauxite and graphite particles, the matrix alloy and a hypereutectic Al-18Si alloy were

Mortensen et al.[25] have considered the infiltration of fibrous as a moving boundary problem. The basic equations governing the process have been substantiated by experimental work on unidirectional infiltration of alumina preforms by Al and Al alloys. The infiltration of fibers initially at a temperature below the liquidus or the melting point of the metal is possible and results in the formation of solid metal between the fibers at the infiltration front. Infiltration kinetics are determined to a large extent by the amount and morphology of this solid metal. The grain size of the matrix and the extent of fibre/matrix interfacial reaction can therefore be largely influenced by controlling the initial preform temperature.

Continuing to their work Mortensen et al. [26,27] have derived a general expression to describe fluid flow and heat transfer during infiltration of fibrous preforms by pure metal. Analytical solutions to the problem are given for the case of unidirectional infiltration into a uniform preform of aligned fibres under constant applied pressure.

Calculations are carried out for infiltration kinetics, using alumina fibre aluminium composites as an example .

In the case of fibres at temperature significantly below the metal melting point, the factor most strongly influencing infiltration is the solidification of metal in the interfibre region. It is assumed that this solidification is in the form of uniform solid metal sheath around the fibers. Metal superheat, when present, serves to progressively remelt the solidified sheath from the up stream end of the preform.

Fibre volume fraction and initial temperature are predicted to have major effect on infiltration kinetics, while metal superheat exert relatively minor influence. when no external heat extraction is present, and



a constant pressure is applied to the metal, flow through the preform continues indefinitely. For the case of external heat extraction flow ceases when sufficient solidification occurs to block flow.

Finally they have presented experimental data to test the results of the theory for pure metal flowing into fibrous alumina preforms.

An apparatus was designed and built for unidirectional infiltration under constant pressure and carefully controlled temperature. A sensor was developed to measure the position of the liquid metal in the fibrous preform during the experiment. Experimental data are reported for infiltration by 99.999 % and 99.9 % wt. pure Aluminum of alumina fibers fabricated into two-dimensionally random preforms. Fibre volume fraction was varied from 22 % to 26%, fibre preheat temperature was varied from approximation 483 to 743k, and metal superheat was varied from 20k to 185k. Infiltration pressure was varied from 1 to 4.5 Mpa (145 to 650psi). An agreement between theory and experiment was very good under all the experimental conditions studied for the 99.999 % wt. pure matrix. The impurity level of the metal influences infiltration significantly. The measured permeability for 99.9 % Al was lower than that for 99.999 % aluminum.

Michund et al. [28] have developed a theoretical analysis to describe the infiltration of fiber preforms by a binary alloy, and its solution was given for unidirectional adiabatic infiltration under constant applied pressure, they further developed the analysis by proposing a model to predict the permeability of fibrous preforms containing solidified primary metal, by deducing the final composite microstructure from processing parameters and by addressing the influence of external cooling on macrosegregation within the composite. Experimental procedures established for

the infiltration of the fibre preforms by pure aluminum are modified to produce samples infiltrated under nearly adiabatic conditions.

Samples of alumina fiber preforms infiltrated adiabatically under constant applied pressure with Al-4.5 wt pct Cu show longitudinal variations in copper concentration, which are well predicted by the theory presented. The microstructure in the infiltrated composites samples also agree with their analysis : the grain size is small where solid and liquid matrix coexisted during infiltration, whereas it is large when remelting occurred indicating that alumina fibers do not promote nucleation of Al-Cu. The model proposed for permeability of the preforms in the presence of solidified metal yields infiltration rates in agreement with experimental data. Finally samples produced under non-adiabatic conditions exhibit transverse macrosegregation; this was explained for simple limiting cases of heat transfer at the die wall.

Jarry et al. [29] further investigated preforms of 20 Vf alumina fibers infiltrated with Al-4.4 wt pct Cu-0.3wt pct Mg using horizontal die casting machine. Fiber preform temperature is varied from 673 to 973k. Solute distribution, fibre volume fraction, and matrix microstructure are characterized using optical metallography and electron microprobe analysis.

Increase in fiber volume fractions are observed in the composites downstream of the infiltration path. They proposed that these resulted from locking of the compressed fibre by solid metal presented during infiltration. With this assumption they found good agreement between theory and experimental data presented in the previous works for solute concentration, fibre volume fraction distributions, as well as matrix microstructure. With an initial preform temperature of 673k, freckles were found in

the composite, which were suggested to result from the combined effect of pressure and significant enrichment in solute at the infiltration front.

Melander et al. [12] have investigated metal matrix composites manufactured by infiltration squeeze casting technique. The matrix was based on an Al-2% Mg alloy and the fibre preform contained 10% alumina fibres. The fibre distribution of the composite was studied by optical Image analysis, this included characterization of the anisotropy of the fibre orientation, volume fraction, and size distribution. Mechanical testing was performed on the composite and the results were compared to tests on squeezed cast material based on the alloy only. The composite had significantly higher proof stress (factor of 2) and ultimate tensile strength (factor of 1.5) than the alloy. Fatigue tests were performed in a four points bend mode on the composite and the alloy. The nominal fatigue strength of the alloy was found to be slightly higher than that of the composite. The stress amplitude was, however, estimated based on linear beam theory, which probably over-estimates the amplitude for the alloy than for the composite. Fatigue crack initiation and early growth was studied in interrupted test for the composite. Cracks initiated within fibres and at fiber interfaces. The growth rate was analyzed in terms of the nominal range of stress intensity. It was found that the short cracks grow at an irregular rate due to interaction with the microstructure and that they grow faster than long cracks.

Kim and Lim [30] have fabricated aluminum matrix composite materials containing Sic whiskers and alumina fibers by the direct squeeze infiltration method.

Optimum processing conditions for preforms and squeeze casting are suggested for minimum damage of the reinforcement, the relatively low

pressure of 25 Mpa is applied. Mechanical properties, such as young's modulus and ultimate tensile strength are improved up to 80% by the addition of reinforcement.

As expected, strength of the metal matrix composites, MMCS, decreases with increased testing temperatures. However, these composites have reasonable strength values for high temperature applications. Predictions for elastic modulus and tensile strength of composites fabricated by direct squeeze infiltration method are proposed from the transformed laminate analogy ,and combinations of modified rule of mixtures and shear lag theory, respectively. Proposed predictions show good agreement with experimental data. From fracture surface analyses at room temperature and elevated temperature, it is found that failure mode is ductile on the microstructural level and it becomes more ductile as temperature of testings increases. Based on fracture surface observations and experimental data, strength reductions at elevated temperatures appear to be mainly due to averaging and softening of matrix alloy.

Wang et al.[31] have tested alumina- aluminum matrix composite system through a two body abrasion wear test. The wear resistance of the composite was found to range from 2-6 times that of the unreinforced matrix alloy . A transition in wear behaviour was found for the various sizes of the abrasion particles. With small particles , the wear resistance of the composite increased with increasing the fibre volume fraction, while with large particles wear resistance decreased with increasing fibre content .The effect of abrasive particle size on this transition may be explained by theoretical analysis of the the fracture of brittle in a ductile matrix under point loading.

Clegg et al. [32] have studied the tensile deformation and fracture of

Al alloy-composite containing delta alumina fibers . They measured the internal stress and showed that the tensile behaviour of these materials is determined by the development of these stresses due to the plastic flow of the material, differences in elastic constants of the two phases and residual thermal stresses were developed during fabrication. Fracture is observed to occur by growth of cracks from failed fibers until a crack large enough to nucleate catastrophic failure is formed.

Manohran et al. [33] have studied cast and extruded aluminum alloy based particulate metal matrix composites. The matrix was 6061 Al alloy and reinforcement consisted of 15 % volume fraction of alumina particulate . The composite was solution treated at 520 C for 4 hours and artificially aged at 175 C for 100 hours to produce an over aged microstructure .

Fracture toughness tests were conducted on fatigue precracked three point bend specimens. From uniaxial tensile tests the following tensile properties were determined, yield strength 285 Mpa, ultimate tensile strength 325 Mpa and reduction in area 16% .Microcracking occurred ahead of the crack tip as the crack progressed. These microcracks were primarily associated with cracks in the alumina particles, and as the the crack progresses the region of plastically deformed material the associated microcracks remain in the wake of the crack.

Baxter et al. [34] have proposed a method to calculate the strength of metal matrix composites reinforced by discontinuous fibers. They showed that the fibers oriented perpendicular to the stress distribution play the key role and the strength depends upon the strength of interfacial bond. If the bond strength is larger than the matrix strength the composite strength has a maximum value which increases with  $V_f$ , and If the bond

strength is weaker than the matrix strength the composite strength has a minimum value which is either weakly dependent or even independent on  $V_f$ . Upper and lower limits of composite strength were calculated and found to be in good agreement with examples taken from the literature of aluminum composites reinforced with either alumina, graphite, or sic .The strength of the matrix alloy is shown to be of very important parameter, weak alloys are easily strengthened, while in certain cases , strong alloys may be weakened.

## 2.4 Significance of The study

It can be seen from the previous literature survey that metal matrix composites is a new field in composite material technology, and is not fully established. Therefore, further research and studies particularly in fabrication techniques of the particulate composites, and examinations of their microstructure, mechanical behaviour and formability are needed.

Little research has been conducted on fabrication of AL- $AL_2O_3$  metal matrix particulate composites by means of vortex technique and still some of the parameters such as rate of addition, mixing speed, mixing angle, and incorporation mechanism need to be investigated.

These formed the main objectives of this study.

## Chapter 3

# Theoretical Considerations

Cast metal matrix composites are made by introducing fibers or particles into a molten or partially solidified metals, followed by casting these slurries in moulds. The spatial arrangement of the discontinuous phase in the cast structure principally determines the properties of the cast composite.

Until the beginning of 1980's aluminum ceramic particle composites were generally made by conventional powder metallurgy methods [4,10]. Later , attempts have been made to prepare these composites by liquid metallurgy techniques (where the particles are introduced into the metal prior to solidification ). The major difficulties in fabricating aluminum ceramic composites by metal metallurgy techniques as reported in the literature [4,10,13,14] are : the absence of wetting between molten aluminum and ceramic particles ( oxides or carbides) at temperatures used in conventional foundry practice, which results in complete rejection of these particles when added to the melt, and the density difference between aluminum and ceramic particles causing floating, preceptation and segregation of dispersed particles in the melt and in the solidified castings. These two main problems are discussed in the following sections.



### 3.1 Wetting

Wetting is a measure of surface adhesive contact between liquid metal and the second phase, and is measured by the contact angle.

The wetting properties of ceramics by liquid metals are governed by a number of variables, including heat of formation, valence electron concentration in the ceramic phase, interfacial chemical reactions, temperature and contact time. High temperature and long contact times promote melt ceramic wettability due to reactions at the melt /ceramic interface resulting in reduced contact angles. Figure(3.1) shows the dependence of contact angle on the temperature in Al/ $Al_2O_3$  systems [1,2,3].

A number of studies have confirmed that the wetting is very poor between different forms of  $Al_2O_3$  and molten aluminum, at contact angles ranging from  $\pi$  near the melting to  $\pi/3$  at 1800 K. Small changes in fibre or melt composition are unlikely to effect dramatic reduction in these values by a pure physical mechanism, and most expedients aimed at improving the wetting mechanism by stimulating chemical reactions, often by the way of fibre/ particle coating or by alloying additions. However alloying additions are preferable over coating techniques for their simplicity and cost [24].

While artificial coating is cumbersome, alloying additions that can react chemically at the fibre or particle/ melt interface look attractive, for example  $Al_2O_3$  react with several divalent transition metal oxides to form aluminates isostructural with spinel ( $MgO.Al_2O_3$ ) offering the potential for the formation for strong interatomic bonds with both matrix and the fiber or particles. however, it appears that only lithium which forms  $LiO_{2.5}Al_2O_3$  can rapidly stimulate a suitable reaction without requiring oxygen introduction by agitation. Unfortunately the incorporation of

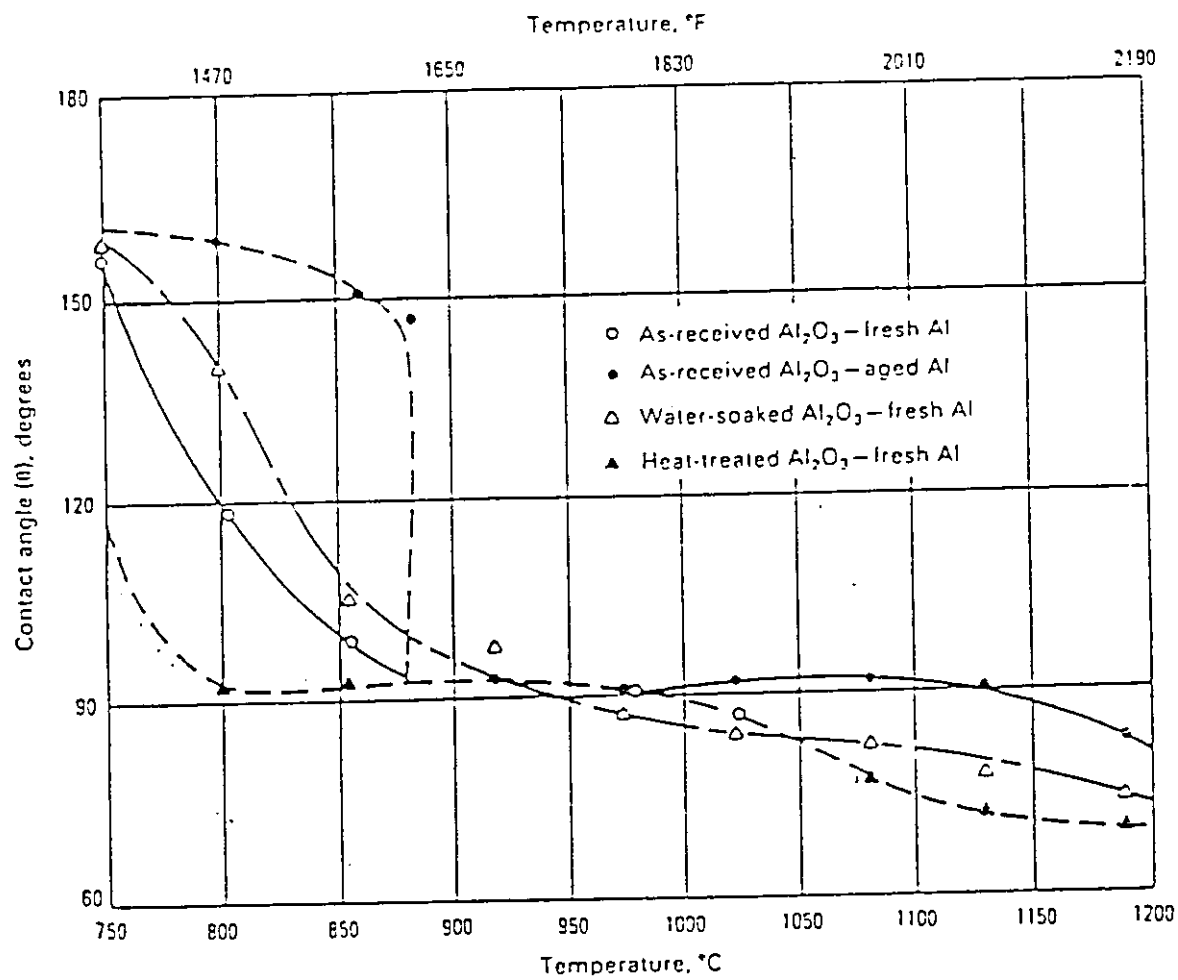


Figure 3.1: Contact Angle in Al/Al<sub>2</sub>O<sub>3</sub> Composite as a Function of Temperature,[1,2,3].

lithium can be associated with certain fabrication and handling problems [17,18].

In the metal Matrix composites, as in their polymer counterparts, the interface bonding can play the key role in determining the mechanical properties of the system. The reason is that, to optimize mechanical properties as stiffness and tensile strength; transfer of load from the matrix to the reinforcing phase is necessary. The thickness and the uniformity of the interfacial reaction layer, Figure(3.2), can be controlled by optimizing melt temperature, residence time of particles or fibers in the melt, and the degree of agitation [1,35].

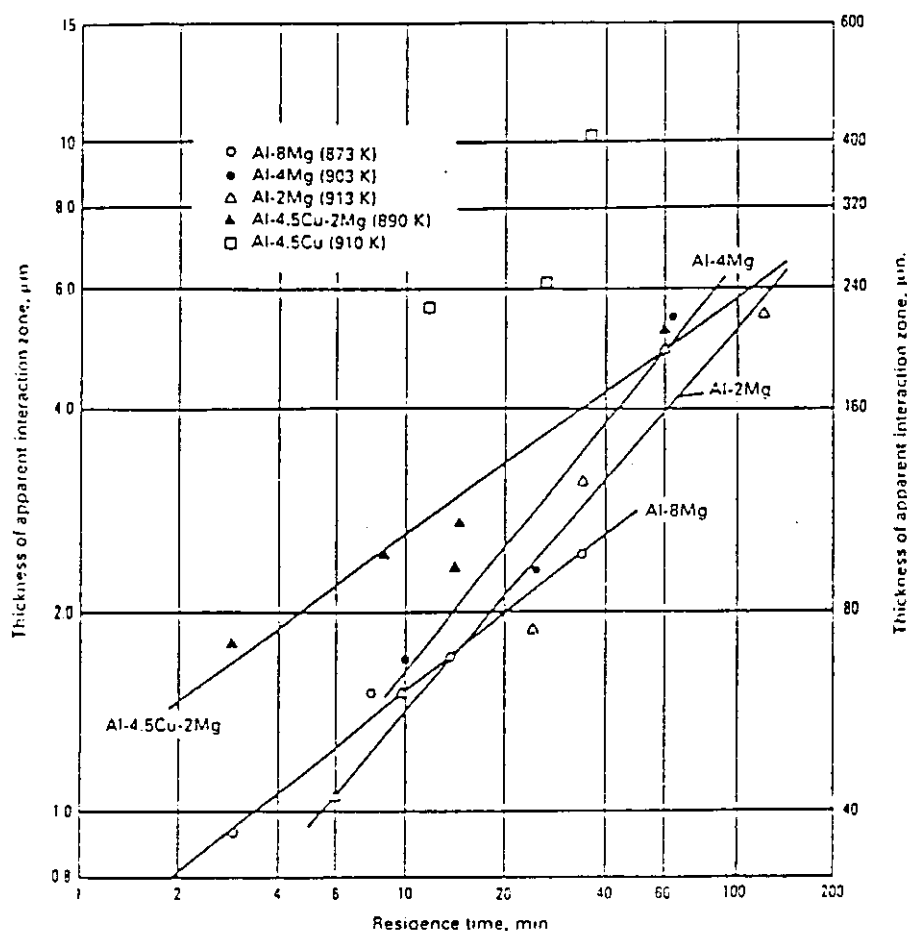


Figure 3.2: Average Thickness of Interaction zone as a Function of Residence Time of  $Al_2O_3$  Fibers,[1,35].

## 3.2 Distribution and Uniformity

When the wetting problem is optimized, the efforts then are concentrated on optimizing the distribution of the particle or fibers (second phase) in the melt by optimizing the governing variables.

The uniformity of particles dispersion in the melt before solidification is controlled by the dynamics of particle movement in agitated vessels. Because of inherent complexity of dispersion, it is difficult to optimize the initial processing conditions to achieve a specific particle distribution in the cast matrix. The distribution of these particles can be controlled by optimizing the following variables [15,16,22,26,27,30]

1. Melt temperature.
2. Residence time of particles in the melt.
3. The degree of melt agitation.
4. Cooling rate.
5. Viscosity of the solidifying melt.
6. Shape, size, and volume fraction of the second phase.
7. Thermal properties of the second phase and matrix alloy.
8. Interactions of crystallizing phase with particles, nucleation of primary phases on ceramics, and entrapment or pushing of second phase by solidifying interfaces.
9. The flocculation (clustering) of particles.
10. The presence of any external forces during solidification.
11. The rate of addition of the particles.

Mixing techniques generally used for introducing and homogeneously dispersing a discontinuous phase in a melt are [1]:

1. Addition of particles to a vigorously agitated fully or partially molten alloy.
2. Centrifugal dispersion of particles in a melt.
3. Dispersion of pellets or briquettes, formed by compressing powders of the base alloy and the ceramic phase, into a mildly agitated melt.
4. Injection of discontinuous phase into the melt with an injection gun.
5. Addition of powders to an electromagnetically stirred melt. The turbulent flow caused by electromagnetic stirring is used to obtain a uniform suspension.

### 3.3 Solidifying Techniques

Among the many techniques available to synthesize metal-matrix composites, solidification processes are particularly attractive for their simplicity, economy and flexibility. Engineering interest in the synthesis of metal matrix composites by casting techniques dates to the 1960s . But a quantitative understanding of the formation of solidification microstructure, which can be used to control solidification processes to achieve tailored properties has emerged only recently.

Quantification has progressed further in the directionally aligned fibre composites made by pressure infiltration of fibre preforms, which facilitates analysis and explanation of experimental observations due to geometrical symmetry and fixed stacking of fibres. In the case of particulate composites, especially those made by stirr casting, where the particles are free to move, the quantification of structural features that emerge during solidification is limited, although the basis elements and variables that influence structure formation in selected systems are catalogued and qualitatively understood . From a phenomenological stand point, the solidification studies on composites have evolved as an independent discipline within the larger frame work of solidification science [35].

There are various techniques used to solidify melt particle slurries , the choice of casting techniques and mould configuration is of prime importance to the quality (soundness, particle distribution) of a composite casting because the suspended particles experience buoyancy - driven movement in the solidifying melt until they are encapsulated in the solidifying structure by crystallizing phase , Particles , such as graphite , mica, talc porous alumina , and hollow microballoons are lighter than most

aluminum alloys and tend to segregate near the top portion of gravity castings , leaving behind a particle impoverished region near the bottom of casting similarly , behaviour particles such as zircon , glass ,  $Al_2O_3$  ,SiC ,SiO<sub>2</sub>, TiO<sub>2</sub>,ZrO<sub>2</sub> tend to settle and segregate near the bottom portion of gravity casting [36].

The various techniques used to solidify melt-particle slurries are discussed bellow:

### 1. Sand casting

The slow solidification rates obtained in the sand mould permit considerable buoyancy -driven segregation of particles . Depending on the intrinsic hardness of the dispersed particles , high volume fraction surfaces can serve as selectively reinforced surfaces -for example abrasion resistant contacting surfaces for various tribological applications [1].

### 2. Die casting

The relatively rapid solidification rates in metallic moulds generally give rise to a more homogeneous distribution of particles in the cast matrix .

Further improvements in particle distribution can be achieved by using water-cooled moulds,copper chills [1].

### 3. Pressure filtration techniques

Pressure filtration or squeeze casting or liquid forging of metal matrix composites is a process that involves unidirectional pressure infiltration of fiber preforms or particle beds by a melt in order to produce void free , near net shape castings or composites, Figure(2.3). The squeeze casting of composite melts into finished shapes

promotes a fine equiaxed grain structure because of large undercoolings and rapid heat extraction[34].

#### 4. Centrifugal casting

Centrifugal casting of composite melts containing particle dispersion results in the formation of two distinct zones in the solidified material: A particle rich zone and a particle - impoverished zone. If the particles are lighter than the melt ( for example graphite , mica, or carbon microballoons in the aluminum , the particle-rich zone forms at the inner circumference, whereas the outer zone is particle rich zone if the particles are denser than the melt ( for example zircon , $Al_2O_3$ , or silicon carbide in aluminum) [1,4].

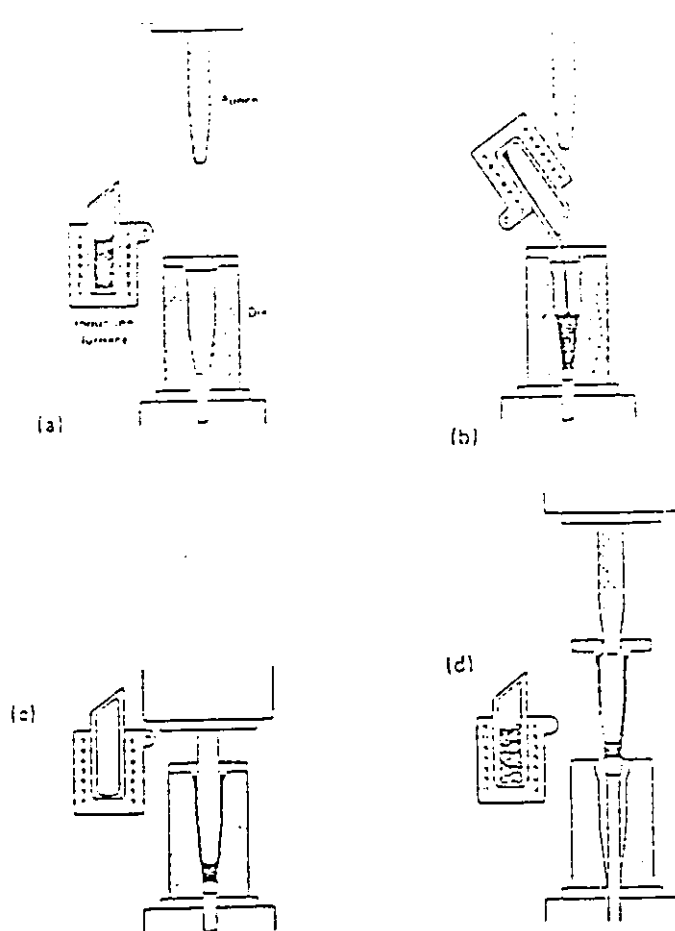


## 5. Vortex technique

In this technique the particles or fibers are added to a vigorously agitated molten metal and then are casted in the mould [4]. Here the vortex is created by mechanical action i.e mixing by impeller, while in centrifugal technique the vortex is created by centrifugal force.

## 6. Compcasting ( Rheocasting)

Particles or fibers of ceramics have been incorporated into vigorously agitated , partially solidified aluminum alloy slurries by the compcasting techniques. The discontinues ceramic phase is mechanically entrapped between the proeutctic phase present in the alloy slurry, which is held between it's liquidus and solidus temperatures. This semifusion process allows near-net shape fabrication by extrusion or forging because deformation resistance is considerably reduced due to the semifused state of composite slurry [1,27].



Schematic illustrating squeeze casting process operations. (a) Melt charge, preheat, and lubricate tooling. (b) Transfer melt into die cavity. (c) Close tooling, solidify melt under pressure. (d) Eject casting, clean dies, charge melt stock.

Figure 3.3: Squeeze Casting Technique [1].

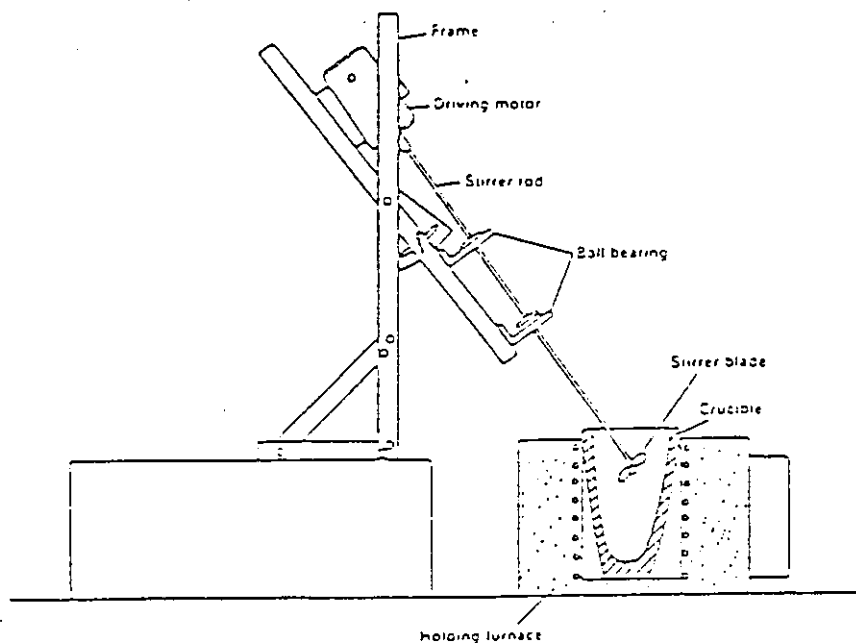


Figure 3.4: Vortex Technique.

## Chapter 4

# Materials, Equipment and Experimental Procedure

### 4.1 Materials

The materials used in this research include:

1. Commercially pure aluminium with a minimum purity of 99.95% supplied by ARAL.
2. Alumina with particle size range of 100 – 200  $\mu\text{m}$ , SG = 4.0 supplied by Fluka Switzerland.
3. Titanium supplied as rich titanium–aluminium master alloy.
4. Methanol (C.P. = 96 %).
5. Diamond paste for polishing (  $\mu\text{m}$ ).
6. Graphite rods 150 mm long, used for mixing.
7. Killer etching solution.

### 4.2 Equipment

1. Voss mixer model 2020, England with a speed range of 50 – 3000 rpm (illustrated in Figure(5.10)).
2. Dual action shaker, model 3508, (illustrated in Figure(5.7)).

3. Multi point digital thermometer (Microprocessor thermometer 6200).
4. Microscope.
5. Universal testing machine, Instron model 1195 of 25 KN capacity was used to determine the mechanical behaviour.
6. Grinder.
7. Polisher.
8. Digital balance.
9. CNC turning machine and carbide tools were applied for machinability tests.
10. Tele surf 10 to measure surface roughness.
11. Pin and disk assembly for determining wear characteristics.
12. Brunell hardness test machine.
13. Silica pipe, 300 mm in length and 20 mm in diameter was used for injecting the powder to the molten metal.
14. Stainless steel crucible (150 mm ID, 200 mm OD) made of 4 mm thick stainless steel hollow pipes, filled by Sic powder in between.
15. Stainless steel moulds (30 mm diameter, 100 mm in hight) were used in melt casting.
16. SiC-Crucibles.

## 4.3 Experimental Procedure

### 4.3.1 Wetting Optimization

At the beginning of the experimental program, it was observed that, adding the as-received alumina to the molten aluminium resulted in rejection of the powder by the melt, so heat treatment procedure was followed to promote wetting by preheating the  $Al_2O_3$  powder, 50 grams of  $Al_2O_3$  powder were preheated for a range of temperatures 250 to 1750 °C for a duration of one hour and mixed with 250 gms of molten aluminium at 850 °C using graphite rods -graphite is non wettable by molten aluminium- followed by casting the composite in water cooled stainless steel mould of 30 mm ID and 100 mm height. For each selected preheating temperature, two samples were taken from the slurry and prepared for metallurgical examination, following the procedure explained later in this chapter. Finally, the optimum preheating temperature of the powder is adopted throughout this work (c.f. Figure(5.1)).

### 4.3.2 Fabrication of Al/ $Al_2O_3$ Metal Matrix Particulate Reinforced Composite

Different techniques were tried to fabricate the Al/ $Al_2O_3$  metal matrix particulate reinforced composite, these are:

#### 4.3.2.A Injection Mixing Technique

In this technique 500 gm of pure aluminium were melted in Si crucible at 850 °C , afterwhich an initially preheated 250 gm of  $Al_2O_3$  powder were injected by means of silica pipe 20 mm below the surface of the melt using pressurized air.

The pressure of the air used for injecting the powder was regulated using a valve fitted to the air main supply, pressure regulation was such

that it permitted the injection the powder without causing excessive bubbling in the melt. After injection of the powder, the slurry composite is mixed by means of graphite rods before casting. Finally, the mix is casted in water cooled stainless steel moulds. Specimens were prepared from the cast for metallurgical examinations.

An important factor which was found to affect the mixing process, is the tip diameter of the silica pipe used for injection of the  $Al_2O_3$  powder. Therefore, the tip diameter was varied from 1 to 10 mm to determine optimum diameter at the same working conditions, and each time the cast was obtained, following the same procedure, two specimen of each cast were prepared and examined metallurgically, to find the most appropriate diameter of silica pipe tip.

#### 4.3.2.B Centrifugal Mixing Technique

The Sic crucible was placed in position on the base of the centrifugal mixer and fastened by means of top sliding plate held fixed by two nuts on two long screwed rods as illustrated in Figure(5.7), a 20 mm thick asbestos insulation layer was inserted between the crucible and centrifugal mixer to protect it from the heat transferred from the molten aluminium in the Sic crucible.

500 gm of Al were placed in a Sic crucible and heated to 950 °C to allow for cooling to 850 °C during the time required to place the crucible and fasten it in position on the centrifugal mixer. 250 gm of preheated powder were added to the melt to the vortex wall created by the centrifugal action, after the incorporation of the alumina powder is completed the Sic crucible is exposed to forced convection by air cooling by means

of pressurized air so as not to give the wetted particles enough time to segregate to the bottom of the crucible. In the second part of this technique, the same procedure and quantities are followed but the preheated alumina powder particles were incorporated by injecting them just below the vortex wall. Finally two specimen are taken from each incorporation technique and prepared for metallurgical examinations.

#### 4.3.2.C Vortex Technique

In this technique, 250 gm of preheated  $Al_2O_3$  powder were added to the vortex of the aluminium melt in the temperature range of 800 – 900 °C, then pouring at the temperature of 800 °C, this temperature was found from preliminary experiments starting from a recommended temperature of 720 °C[4]. To allow solidification without existence of porosity, the temperature was increased from 720 °C, the recommended temperature for  $Al_2O_3$  powder and then examining the cast for porosity existence. The temperature was increased in steps of 50 °C up to 820 °C when it was found that the cast was completely free from any porosity, then another test was made at 800 °C and the cast was also found to be free from any porosity. Hence this temperature was adopted as a pouring temperature for the slurry .

Since the vortex diameter varies with the melt temperature and vortex speed, it was necessary to establish the relation between the parameters for the impeller used in the experiments to inject the powder at the proper time to achieve a cast free of defects. The first was to determine the cooling rate of the molten aluminium vortex at a particular speed. This was achieved by inserting a thermocouple just below the surface and

measuring the temperature drop with time for the melt from 1000 °C to 800 °C, the readings were taken every 10 seconds using a stop watch. The cooling rate was found to be 5 °C/min .

Then an experimental relations was established between the vortex diameter, the melt temperature and impeller speed. This relation is shown in Figure(5.3), every point in this figure is the average of ten readings, it can be seen that the ratio  $D_v/d = 5/3$  is optimum as it caused wetting most of the preheated powder particles and required minimum time than the other ratios (4/3 and 7/3). The ratio 4/3 needs more time to wet all the added powder, whereas the ratio 7/3 retards the powder to the crucible edges. In each case the slurry was casted in water cooled stainless steel mould.

Two methods were tried in injecting the preheated powder to the melt first, the preheated powder was directly added to melt at the vortex wall, and the second by injecting the powder just below the surface of vortex wall and each case the composite slurry was casted in a water cooled stainless steel mould. Specimens were prepared from each case for metallurgical examinations.

#### 4.3.2.D Compcasting Technique

A quantity of 600 gm of preheated powder is added to 500 gm of partially solidified molten aluminium at 650 °C, at an impeller running speed of 700 rpm in the stainless steel crucible, the mixer is moved back and forth during the addition of the powder.

In this technique the mechanical shear action of the impeller in the partially solidified aluminium is seen to be efficient in wetting all the added powder the rich slurry is similarly casted in air cooled stainless



steel moulds. Samples are taken and prepared and prepared for metallurgical examinations.

#### 4.3.2.E Modified Vortex Technique

In this technique a rich slurry is prepared by the compocasting technique discussed in the previous section, and then the rich slurry is added to a quantity of molten aluminium at 950 °C and mixed by means of an impeller. If the fluidity of the slurry is not enough to allow pouring, then more Al at 950 °C is added to the mixed slurry.

When the temperature of the composite slurry reaches about 800 °C the slurry is casted in the water cooled stainless steel mould. Similarly samples are taken from the casted slurry and prepared for metallurgical examinations.

#### 4.3.2.F Modified Vortex Technique with Addition of Titanium as a Grain Refiner

It is well established that titanium can be used as a grain refiner for aluminium, hence improving its fluidity and mechanical behaviour. Therefore it was thought to try it in modified vortex technique described in the previous section. Three percentages by weight; namely 0.05, 0.075 and 0.1 were used. The titanium was added to the aluminium before incorporation of the rich slurry and the same procedure of the previous section was used. Finally the melt slurry was casted in a water cooled stainless steel mould, and specimens are prepared for metallurgical examinations.

## 4.4 Metallurgical Examination

After the specimens were fabricated by any of the different previously methods, two parts 10 mm below top and 10 mm above bottom were sectioned and then the specimen is sectioned longitudinally. The specimen is then ground along the sectioned planes using different grade of emery paper, namely 120, 200, 400 and 600  $\mu\text{m}$  in sequence, with water flow at the grinding spot, after the grinding process is accomplished then specimens are polished at a leather cloth with the aid of diamond paste (1  $\mu\text{m}$ ) with continuous flow of methanol, until mirror surface quality is obtained, the specimen is then cleaned with distilled water and dried. Finally the specimens are etched in Killer's solution for 30 seconds. The etched specimens are then examined under an optical microscope for

- Particle distribution
- Particle bonding and wetting
- Soundness of the specimen

First the specimen is sectioned under microscope in all the of the sectioned planes, and judged for particle distribution, after that the specimen is scanned for any porosity existence and judged for soundness, then selecting different alumina particles in all the sectioned planes, for each particle the periphery of the particle is magnified and observed for any porosity existence and hence the wetting and bonding is judged.

Finally the volume fraction was determined by assuming that the particles of alumina to be a complete sphere, with an average particle size of 150  $\mu\text{m}$ , by considering any area on the composite specimen then counting the alumina particles in that area, the volume fraction is determined

by dividing the area occupied by the particles over all the considered composite area, this gives the volume fraction of that particular spot.

Since the distribution of particles is not uniform, fifteen spots were considered and the volume fraction  $V_f$  is determined for each spot from which the average volume fraction of the fifteen spots was taken and this average is considered to represent the true value of the volume fraction.

Finally photo micrographs were taken at different magnification ratios.

## 4.5 Mechanical Behaviour Tests

The following two mechanical tests were carried out on the fabricated composite by modified vortex technique with grain refinement and on pure aluminium, compression and hardness tests.

The compression test was carried out to determine the mechanical behaviour of the composite. Three compression tests were carried out on both pure Al and composite specimen (10 mm diameter and 10 mm high), the specimen were prepared on CNC lathe machine and then lapped by Emery paper of 1000  $\mu\text{m}$  to the finished precise size. The compression tests carried out on the Instron machine and the resulting load – deflection curves are transformed to their true stress – true strain curves, Figure(5.18) shows the average true stress – true strain curve, where the variation didnot exceed  $\pm 4\%$ .

Brinell hardness tests were also carried out on specimens of 25 mm diameter and 25 mm high of both Al and composite material to allow comparison.

Finally the percent reduction in height of both Al and composite material for same stress value are calculated as indication of ductility.

### 4.5.1 Wear Analysis

Wear tests conducted on alumina reinforced-aluminum composite prepared by the modified technique described previously ( with 80 BHN hardness) were performed on the pin on disc assembly as illustrated in in Figure(4.4 ),the disc is made of DIAMAX cold spray coated stainless-steel with 45 Rc hardness.

The main parameters in these tests are :

1. Specimen mass... (The loss of mass indicates the wear)
2. Time
3. Sliding speed ...(Rotational speed X Radius of rotation, 75 mm)
4. Load.....(Spring deflection X spring constant, 500 N/M)

Six groups of loads (2.5 N,5.0 N. ,7.5 N. ,10.0 N. ,12.5 N. ,15 N.) are fixed for each sliding speed (1.57 M/Sec., 1.96 M/Sec., 2.36 M/Sec., 2.75 M/Sec., 3.14 M/Sec., 3.53 M/Sec. , 3.93 M/Sec.).

### 4.5.2 Mass Loss with Time

The following procedure was carried out for all the specimens each of 15 mm Diameter and 20 mm high:

1. The disc is mounted on the machine and checked for straightness.
2. The rotational speed and spring load are fixed according to charts in Figure(5.32) and Figure(5.33) respectively.
3. The specimen is cleaned and then its weight is measured and recorded using digital balance.
4. The specimen is plugged in its Housing vertically against the disc.

5. The machine is turned on for 15 minutes and then stopped, the specimen is unloaded and its weight is measured to determine the mass loss at every time interval.
6. Steps (3),(4),and (5) are repeated four times for each specimen giving a total duration of 60 minutes for each test.
7. Accumulative mass loss after each time interval is plotted against time for each specimen.
8. Steps (2) through (7) are repeated for other specimens with different wear parameters.

The effect of interrupting the tests to make the necessary mass measurements was investigated by doubling the previously selected time intervals, no significant difference was detected in the recorded measurements conducted using the different time intervals.

The results are shown in Figure(5.22) through Fig(5.28).

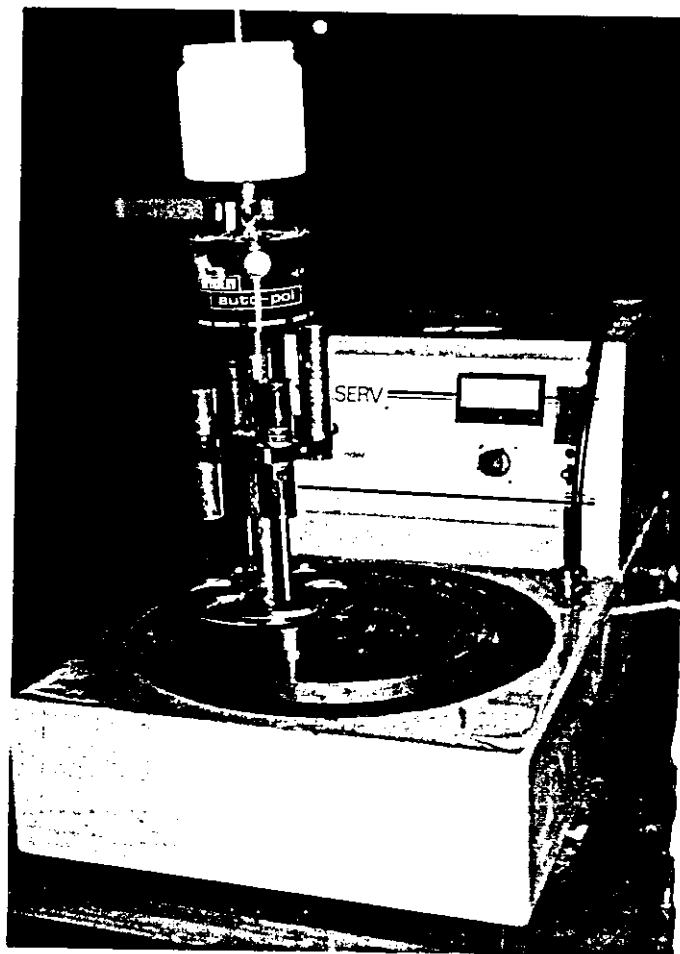


Figure 4.1: Pin on disc assembly

## Chapter 5

# Results and Discussion

### Metallurgical Aspect

Introducing the as-received alumina particles to the molten aluminum at all melt temperatures have proved to be unusefull in retention any fraction of alumina particles ,by all the available techniques , and this comes in agreement with all the previous works survied, the alumina particles were visibly rejected when they were added to the melt at room temperature.

The following variables are to be investigated to obtain the optimum incorporation conditions of the alumina particles to the molten aluminum.

### 5.1 Wetting optimization

According to the literature review, it was found that there is a range of temperatures at which the contact angle of aluminum/alumina composite is minimum , which leads to find out the optimum temperature for this type of alumina particles and aluminum matrix.

After following a long but systematic procedure in relating the amount of retended alumina to the preheating temperature (soaking temperature),it was found that 1050 c" is the optimum temperature , Figure(5.1) shows these results, the preheating process was carried out for one hour

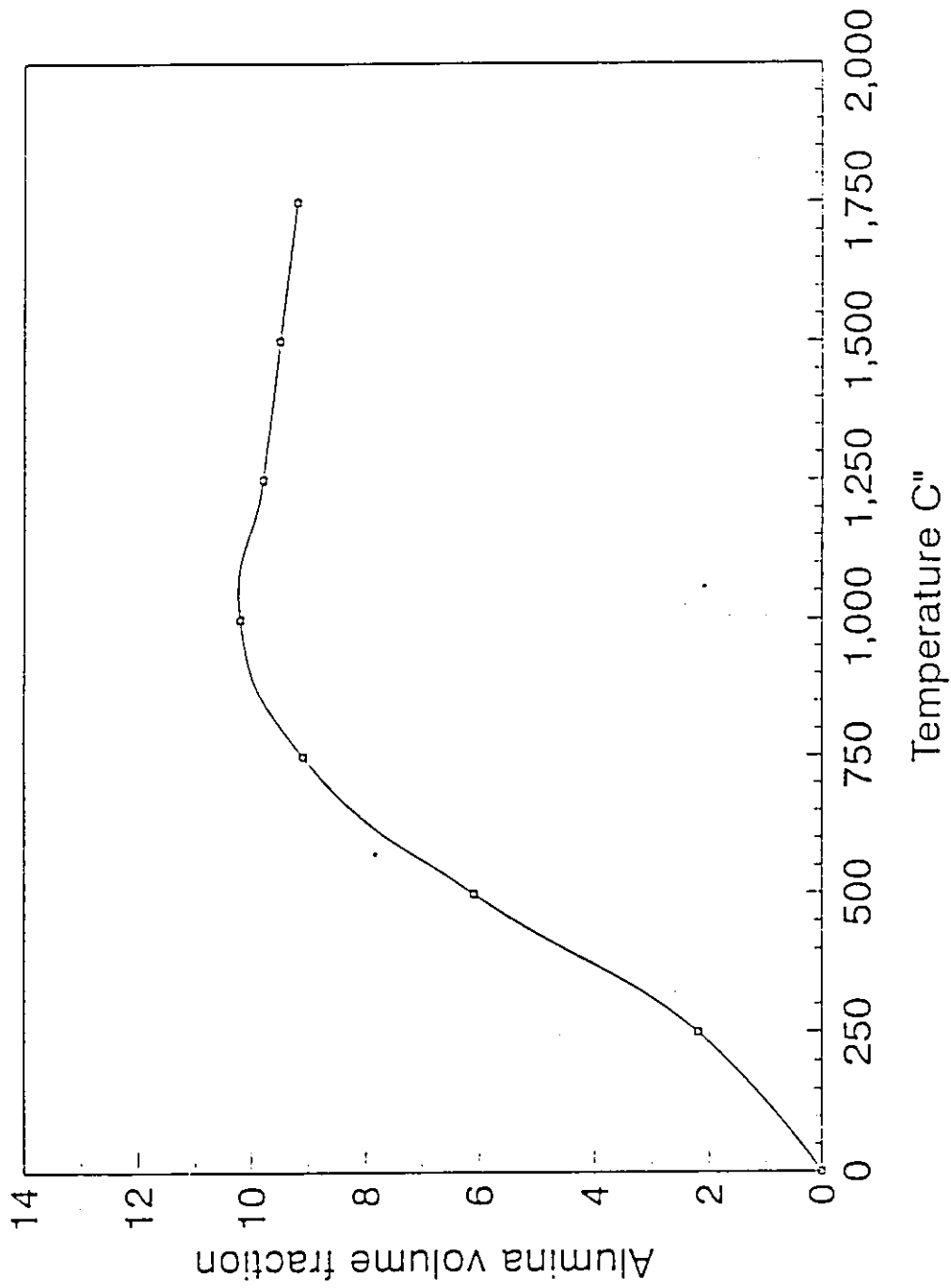


Figure 5.1: Alumina Preheating Temperature Optimization

Figure(5.1) Alumina preheating temperature.



## 5.2 Rate of Addition

It was experimentally found that increasing the rate of addition above 25 gm/min, flocculation and colonies of the ceramic particles were clearly seen at the surface, even though the density of alumina particles is higher than that of aluminum density, and this is due to the fact that the density of the particles colonies is less than aluminum because of incomplete wetting which permits the existence of air bubbles between the particles.

## 5.3 Mixing Speed and Angle of the Impeller

The speed of impeller should fall within a certain range of speeds to create a vortex. This speed should be above a threshold value to initiate open vortex but below a speed which causes the vortex diameter to be larger than the crucible diameter causing the molten aluminum to splash out.

The depth of the vortex is controlled by the immersing depth of the impeller which should be well below the surface of the molten aluminum to open the vortex but not too deep to avoid creating an internal vortex with closed surface, Figure (5.2) shows the vortex parameters.

It was then found that creating the vortex with the impeller at an angle ( $35^{\circ}$ - $45^{\circ}$ ) is more efficient in wetting and mixing the ceramic particles than the case of  $90^{\circ}$ . This is due to minimizing the retarding effect, causing most of particles to slide on the vortex wall and not centrifugally retarded to the vortex edge causing flocculation and clustering to happen.

Actually the crucible shape has a significant importance on optimizing the vortex shape and on the whole process, Figure(5.2).

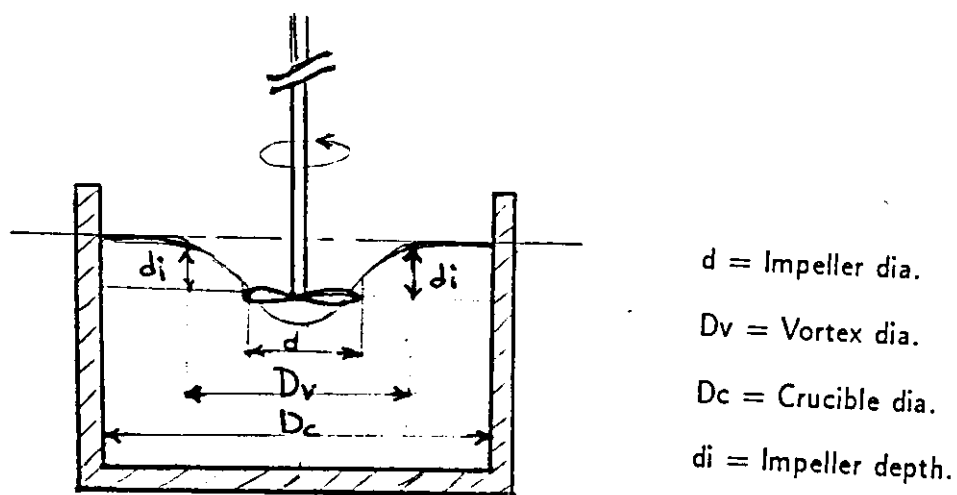
The shape illustrated in Figure(5.2a) is more efficient than the classi-

cal crucible shape Figure(5.2b), in that the surface of molten aluminum is minimized causing the oxidation process of molten aluminum to be minimized thus enhancing the wetting process with minimum edge action.

So one has to accommodate the following factors to create the optimum vortex

1. Temperature of the molten aluminum.
2. Impeller dimension and crucible dimension.
3. Speed of the impeller
4. Immersing depth of the impeller.
5. Angle of the impeller.
6. Crucible shape.

By systematic and lengthy experimental procedure the vortex diameter related to the impeller speed at various holding temperatures was determined and shown in Figure(5.3) Because of limited laboratory facilities in holding the temperature of the molten aluminum, the impeller speed was adjusted to compensate for the temperature drop in order to maintain the required optimum vortex parameters.



### Vortex Parameters

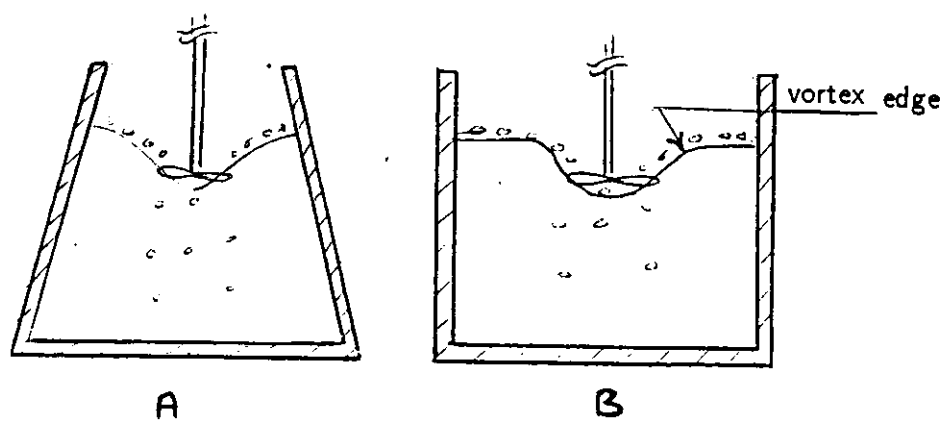


Figure 5.2: Crucible Shape and Its Relation to Wetting Process

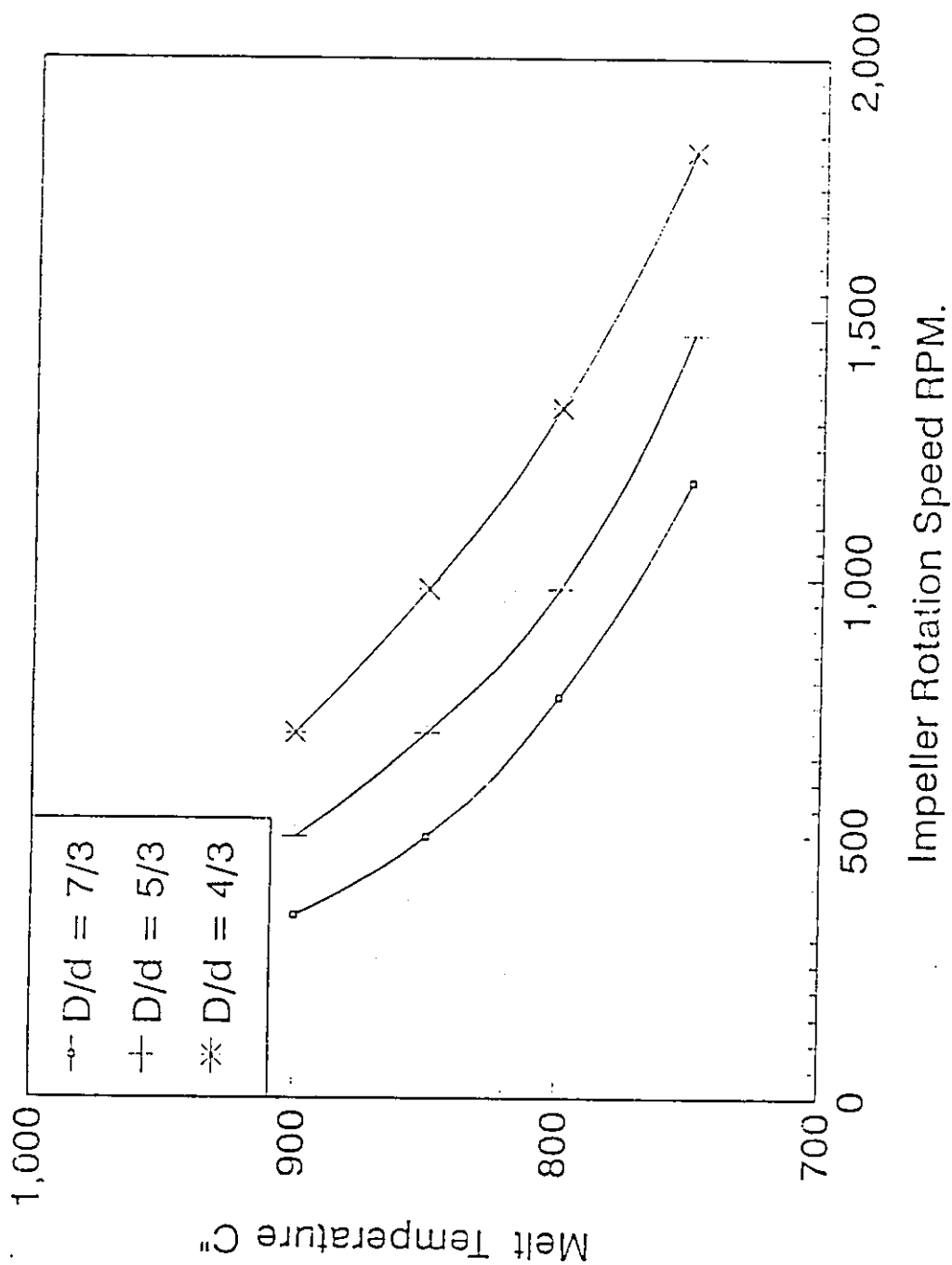


Figure 5.3: Optimum Vortex Parameters

Figure(5.3) Optimum vortex parameters

ject the powder through the molten aluminum but not to cause excessive bubbling through the melt causing the surface to be open and then the particles to be rejected because of air bubbling and oxidation.

The diameter of tip of the silica pipe is of prime importance , if the diameter is larger than 5mm then the particles are injected fastly but in bulcks, which will lead to maximize the clustering and flocculation of particles , and if this diameter is less than 3mm blockage was observed to take place, so a range of 3mm-5mm is considered to be optimum for the powder mesh size used in this work.

The results are illustrated in Figure(5.5), clustering and flocculation are observed, also the distribution is not good, planes near the bottom of the crucible contain larger volume fractions than planes near the surfaces , this is due to the fact that the wetted particles move down and the incompletely wetted particles remain near the surface due to their less apparent density, volume fraction up to 10 % was achieved, also the porosities are noticed with these particles due to surrounding air bubbles between the particles colonies. Figure (5.6) shows porosities and these porosities are due to the particles taken away by grinding process due to the incomplete wetting, resulting in poor bonding.

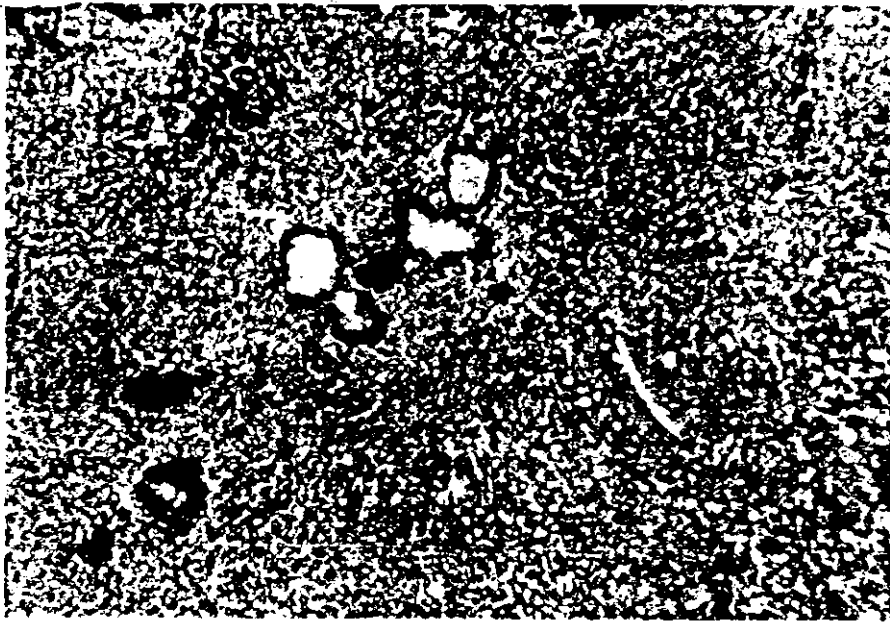


Figure 5.5: Injection Mixing Technique Results, Longitudinal Section, X75

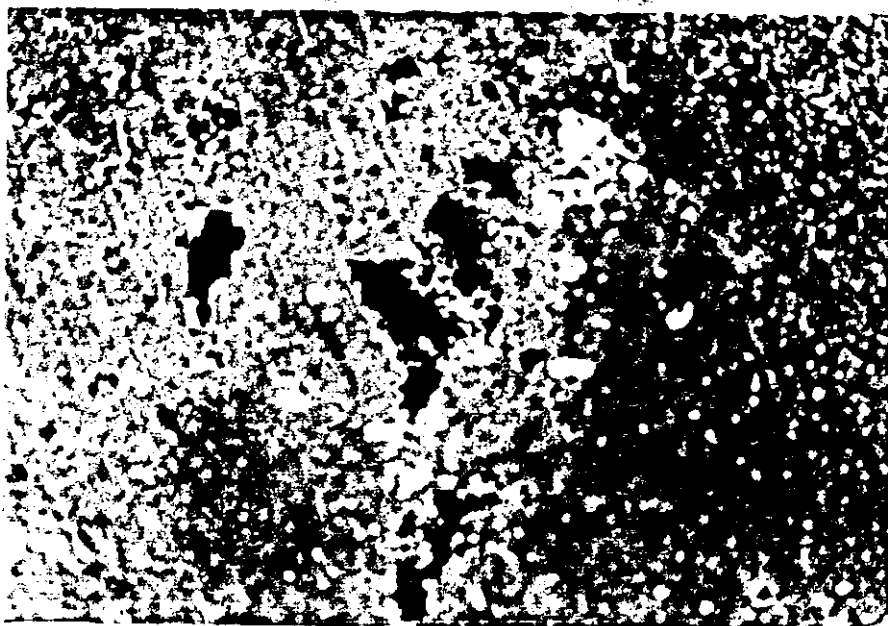


Figure 5.6: Porosities in Injection Mixing Technique, longitudinal Section, X100

little amount will go up the surface and most of particles are distributed away of the center as shown in Figure (5.8). Hence planes near the surface will suffer from porosities, colonies and clustering, but deeper planes are free of colonies, Figure(5.9). All horizontal planes are identical in particle distribution, with a rich zone near the edge and poor zone near the center. Volume fraction up to 10 % was achieved.

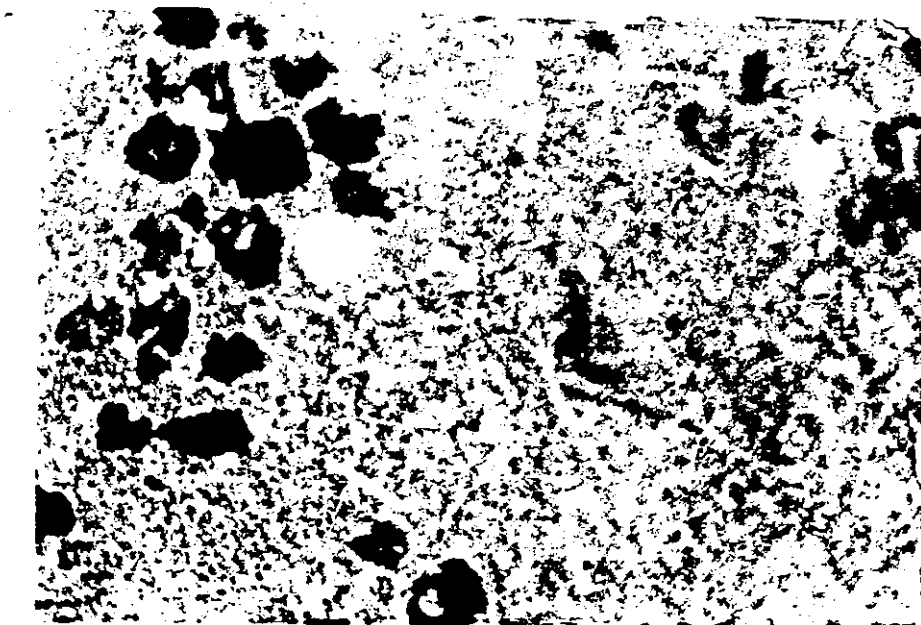


Figure 5.8: Centrifugal Casting Technique Results, Top Section, X100

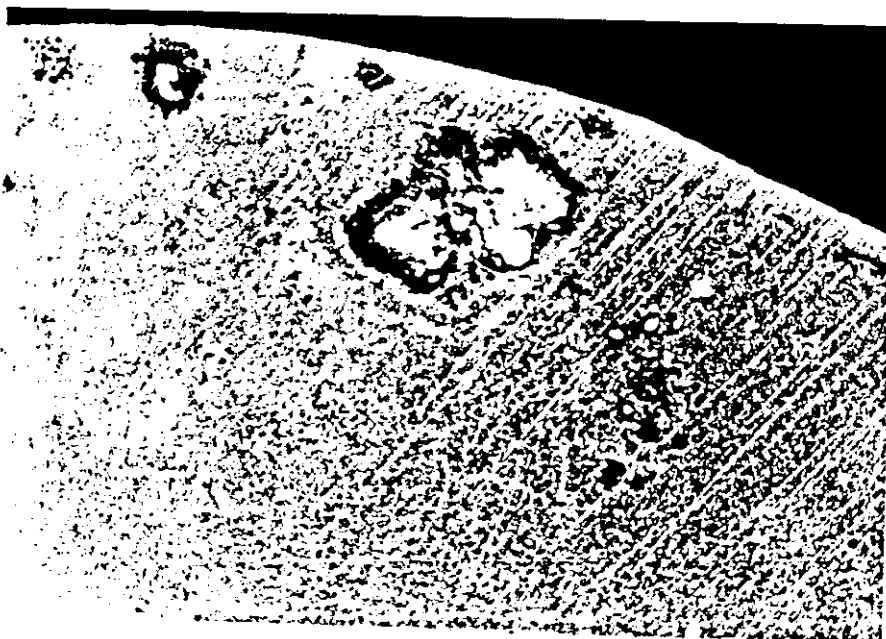


Figure 5.9: Porosities in Centrifugal Casting Technique, Top Section, X150



### 5.4.3 Vortex Technique

In this technique the preheated ceramic particles are added to the molten aluminum on the wall of the vortex created by the impeller as illustrated in Figure(5.10) .

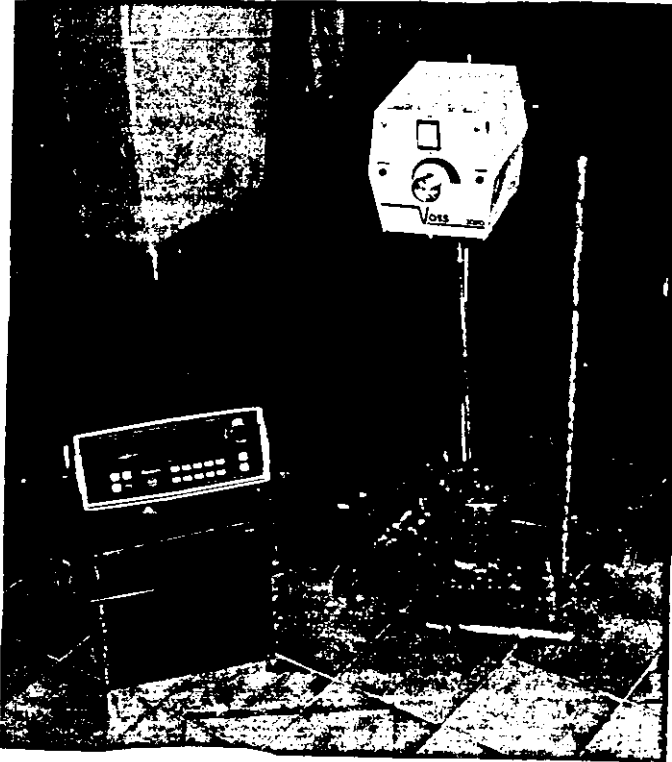


Figure 5.10: Vortex Technique Set up

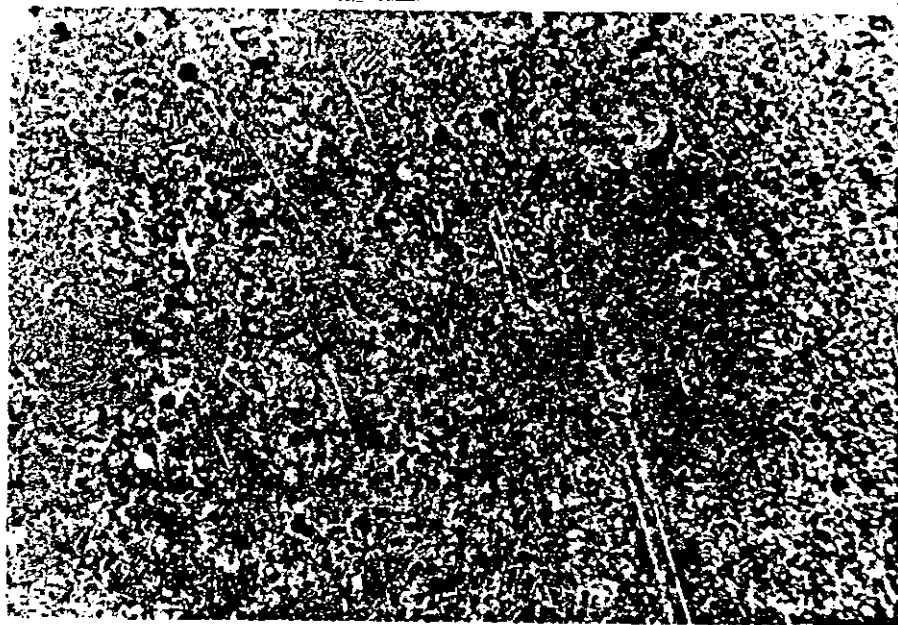
Again the vortex is optimized following the curve developed in section 4.5 , tacking the following values and ranges to the variables and parameters .

1. Mixing speed ranges from 500 rpm to 1000 rpm.
2. Melt temperature between 800 c° to 900 c°.
3. Vortex to impeller diameter ratio, 5/3.
4. Rate of addition, 25 gm/min.
5. Angle of the impeller, 45°.

It was found that adding the particles by injection below the surface of the molten aluminum is more efficient than adding the particles on the vortex wall, and this is due to the fact that the injection below the melt surface makes all the particle surface to be exposed giving better contact with the melt and so enhancing the wetting of particles.

Figure (5.11a) shows different sections of specimen prepared by this method.

By comparing the results of the specimens of this technique to the previous one, one can see the great improvements in the particle distribution along different sections of the specimen, and higher volume fraction of alumina particles, e. g. 25 % at least was achieved. Similarly sections near the bottom of the specimen contain higher volume fractions as seen in Figure (5.11b), because of the gravity action on the particles after complete wetting.

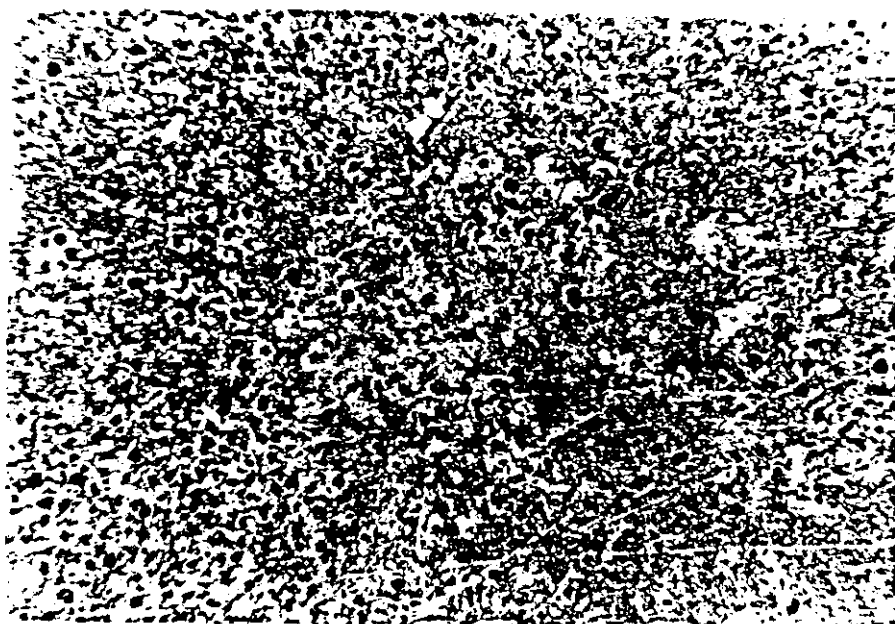


**Top Section**



**Bottom Section**

**Figure 5.11a Vortex Technique Results, X30**



**longitudinal Section, X30**

Figure 5.11<sub>b</sub> Vortex Technique Results

#### 5.4.4 Rheocasting Technique

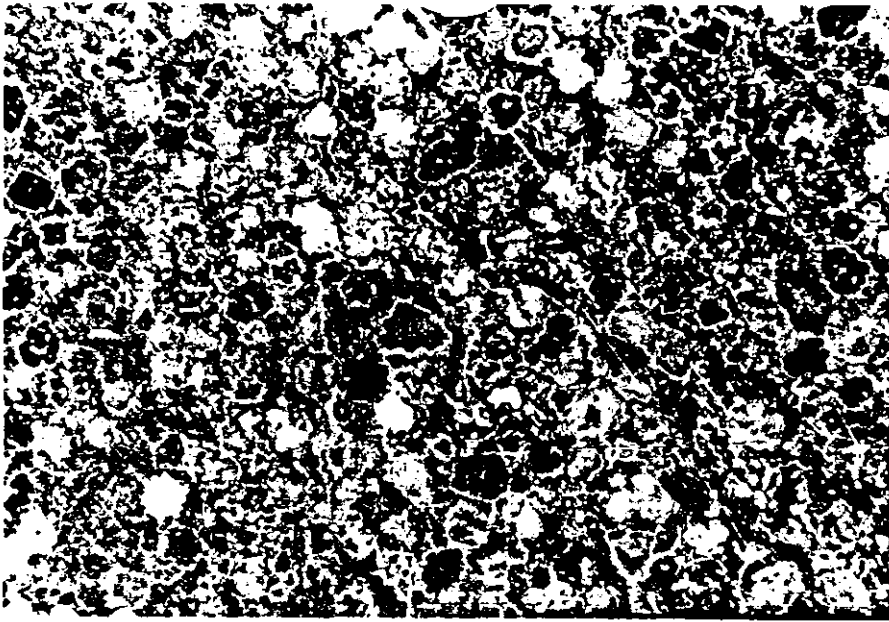
Rheocasting or Compocasting as defined previously in chapter 3 is the addition of the alumina particles to the melt at its liquidus-solidus temperature.

, the well insulated crucible ensures enough time to add all the alumina particles before excessive solidification occurs, the slurry is then casted to its final shape.

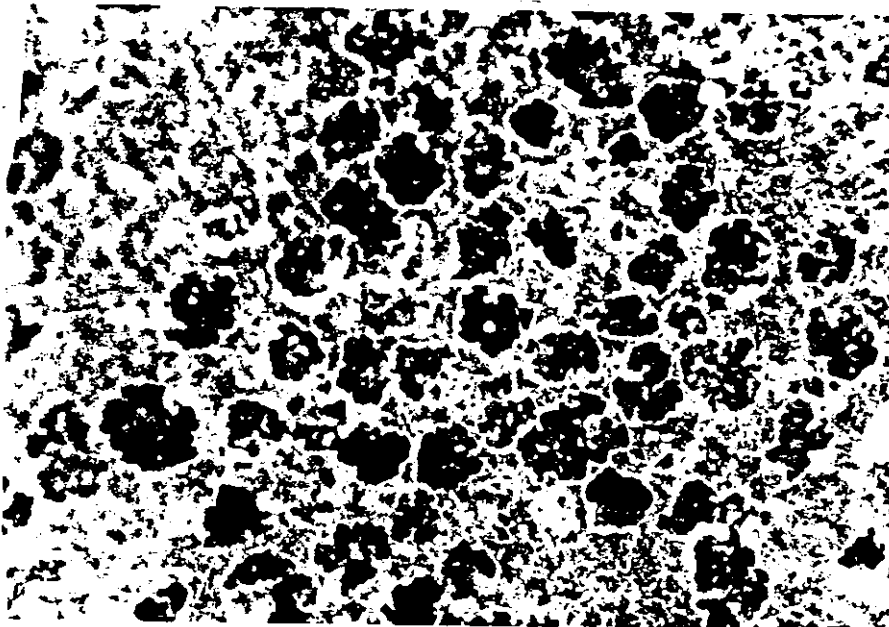
In this method porosities were noticed as a general feature in all the sectional planes of the specimens promoting to the idea of extrusion for these specimens and so imposing limitations on the final shape.

comparing these results with those obtained by vortex technique, better distribution is noticed and higher volume fraction up to 75 % is obtained , Figure (5.12). In fact one can add higher volume fractions of alumina contents but at the expense of soundness of the casts i.e excessive porosity existence.

The large volume fractions and better distribution of alumina particles in this method are attributed to the fact that the viscosity of the melt is less than that of vortex technique, in which the particles are not free to settle down to the bottom of the crucible but they were mechanically distributed by the shear action of the impeller.



**Top Section**



**Bottom Section**

**Figure 5.12: Compocast Technique Results, X95**

#### 5.4.5 The Modified Vortex Technique

Inspecting the previous technique results , one can notice the improvement in certain features (aspects) at the expense of others, e.g. in injection technique poor distribution and significant porosity were noticed. In centrifugal casting specific particle distribution is obtained as a rich zone and a poor zone and so imposing on a specific applications for these cast product. Then in vortex technique more acceptable distribution and complete soundness ( free of porosities) were achieved , but a moderate volume fractions, 25 %, and a significant microsegregation effect were also noticed. Finally in compocasting higher volume fractions, excellent distribution and minimum microsegregation are all achieved but porosities do existence.

Taking all these observations into consideration the following procedure is proposed as a modified method to obtain a sound, minimum microsegregation and porosity with a high ceramic particles volume fractions :

1. A very rich composite slurry is prepared by compocasting technique.
2. Adding this rich slurry to a molten aluminum quantity by vortex technique.
3. Casting the composite melt in a permanent chilled mould.

By following these steps it was possible to join all the advantages of the previous techniques and to avoid their disadvantages. This was possible because of treating the two primary difficulties in MMCs fabrication. In the first step we have treated the wetting problem of large volume fraction of alumina particles by compocasting technique with a minimum microsegregation effect, and in the second step the alumina rich

but porous slurry is diluted in Al matrix by vortex technique to overcome the existence of porosity, the results of this technique are shown in Figure(5.13).

Inspecting these results the following are clearly seen, excellent distribution, high Vf, 50 %, complete soundness, with minimum microsegregation.



#### 5.4.6 Modified Vortex Technique With Grain Refiner

According to the proposals recommended in Chapter 2 we have adopted the titanium as a grain refining agent, adding this agent to the aluminum prior to the incorporation of the Rich alumina slurry was found to be of great effect on minimizing the microsegregation to accepted levels, ie insignificant microsegregation effect.

Microscopic examination revealed that a range of the refining agent of .05% to 0.1 % weight ratio was effective in reducing the microsegregation levels, above this range no further enhancement is detected.

Figures(5.14) through Figure(5.16) inclusive show the results of modified method with grain refining agent of .1% Ti.

These results could be interpreted as follow, as the grain size of the matrix is smaller, better particle distribution is obtained as a result of minimizing the entrapment of particles due to the pushing solidifying interfaces, ie forming a smaller equiaxed matrix grains and less dendritic growth .

**ELECTRO SCANNING TEST**      Figure(5.17) shows electroscanning photo for alumina particle, Complete wetting and hence full bonding are clearly seen.

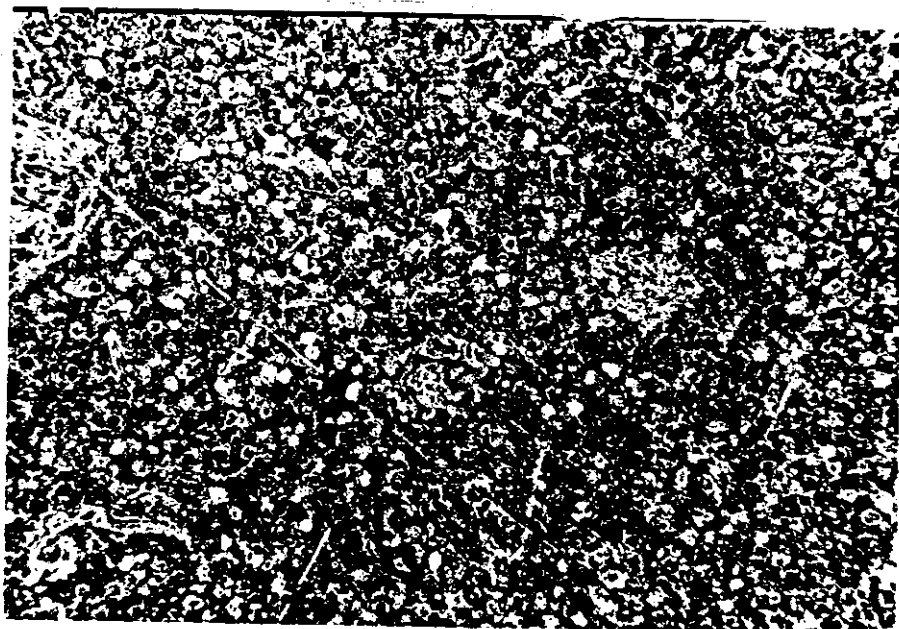


Figure 5.14: Modified Vortex Technique with Refining Agent, Top Section, X25

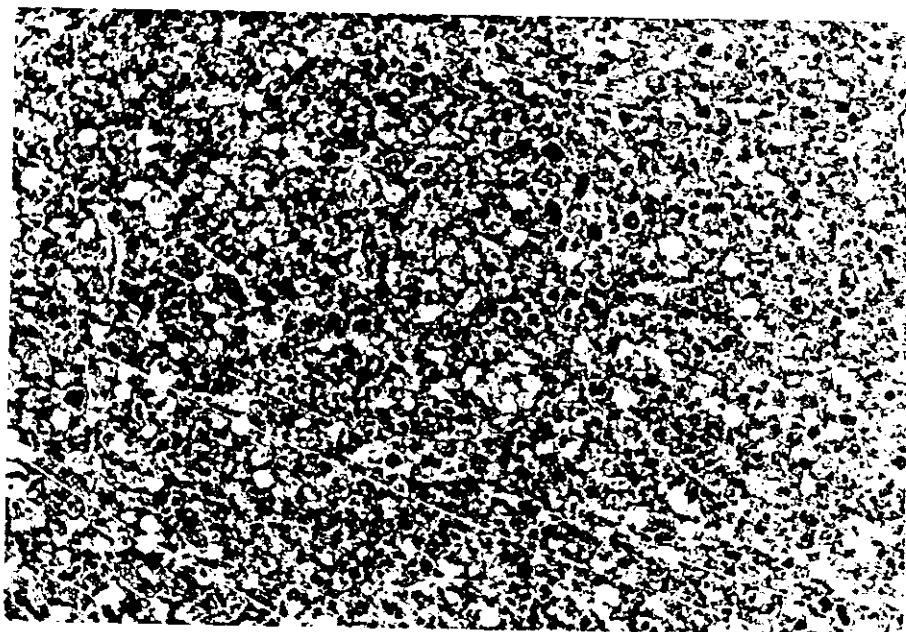


Figure 5.15: Modified Vortex Technique with Refining Agent, Bottom Section, X25

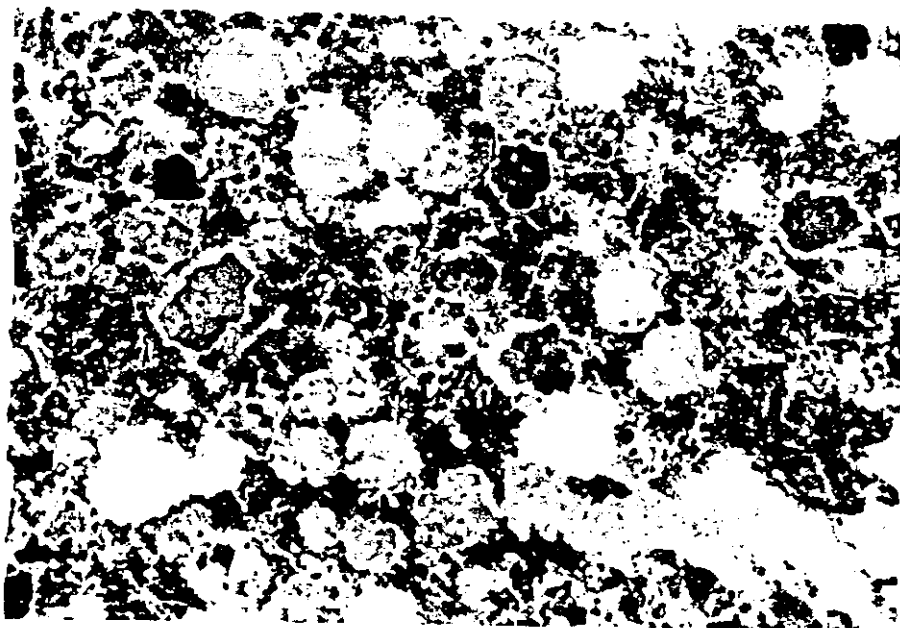


Figure 5.16: Modified Vortex Technique with Refining Agent, Longitudinal Section, X100



Figure 5.17: Electroscanning photo for  $Al_2O_3$  particle

## MECHANICAL ASPECT

### 5.5 Mechanical Behaviour And Hardness Test

Compression tests were conducted on 10 mm dia. and 10 mm high specimens of both pure aluminum and Al<sub>2</sub>O<sub>3</sub> reinforced MMC Aluminum, Figure (5.18) shows the results of mechanical behaviour tests represented as true stress true strain curve, 6-E, it is clearly seen that the addition of Al<sub>2</sub>O<sub>3</sub> particles to the pure aluminum enhances the mechanical properties of pure Aluminum, Ultimate strength is increased from 120 Mpa for pure Aluminum to 240 Mpa the composite material. However the maximum reduction percent in height, i. e. indication of ductility was reduced from 80 % for pure aluminum to 30 % for the composite material.

Hardness tests were performed on both pure and Al<sub>2</sub>O<sub>3</sub> reinforced Al specimens, the BHN is increased from 55 BHN for pure Aluminum to 80 BHN for the Composite.

### 5.6 Machinability

Specimens of both pure aluminum and alumina reinforced aluminum matrix (20mm dia. and 50mm length) were machined on the CNC lathe machine at different cutting conditions to assess and compare their machinability.

The following parameters were varied :

1. Depth of cut, D, in mm.
2. Feed rate, f, in mm/min.
3. Sliding speed, V<sub>s</sub>, in m/Sec.

The results are shown in Figure (5.19) - Figure(5.21) inclusive. The Ra reading shown is the average of 10 different located readings, which give indication of surface quality.

The figures show clearly the enhancement of surface roughness of  $Al_2O_3$  particulate composite over pure aluminum and this is due to that the hardness of the composite is higher than pure aluminum. The cutting action resulting in material removed was associated with small fragments discontinuous chips, while for pure aluminum, where appreciable ductility exists, cutting action is associated with large plastic deformation resulting in large continuous chips, and at some cutting conditions, and at some cutting conditions, the chip was found to remain in contact with the workpiece, and didnot break off. where the cutting tool cannot cut, plastic deformation occurs without material removal.

Figure (5.19) shows that by increasing the feed rate the surface roughness increases,causing low surface quality. Similarly, Figure (5.20) shows that increasing the depth of cut cause increase in the surface roughness.

Figure (5.21) shows that by increasing the sliding speed up to a certain value (4.5 m/sec.) surface roughness decreases, and beyond this sliding speed the roughness approaches a stable value 4.4  $\mu\text{m}$  for pure aluminum and 3.3  $\mu\text{m}$  for the composite material, This is explained in terms of strain rate in the cutting process; as the cutting speed increases, the strain rate increases, resulting in less ductility which changes the material from semi-ductile to almost brittle, hence the chip is of small size resulting in good surface quality. After this speed, the strain rate will have little or no effect.

## 5.7 Wear Analysis

### 5.7.1 Wear Rate Vs. Spring load and Wear rate Vs. Sliding Speed

The wear is plotted against the applied load as shown in Figure (5.29), generally the wear rate increases with increasing the applied load at any sliding speed, except for any speed value within [2.75 - 3.93]m/sec. the wear rate is decreased as the load increased within [2.5-7.5] N range.

Figure(5.30) shows the wear rate against the sliding speed, here the wear rate is increased with increasing the sliding speed at any load value ,except for any load within [2.5-7.5] N the wear rate decreases as the sliding speed is increased within [1.57 -2.36] N.

By summing up the two curves one can clearly notice the following two regions:

Region one: [2.5 N - 7.5 N.] and [2.75 M/Sec to 3.93 M/Sec.]

. The wear rate decrease with increasing the load at any specified value of this velocity range.

Region two: [1.57 M/Sec. to 2.36 M/Sec.] and [2.5 N. to 7.5 N.]

. The wear rate decrease with increasing the sliding speed at any specified value of this load range.

Joseph C. et al. [37] have proposed combined wear mechanism in their explanation of dry sliding wear tests conducted on SIALON Ceramic specimens. This combined wear mechanism involves brittle fracture and plastic deformation, which depends on the relative magnitudes of contact load and sliding speed respectively.

Comparing wear parameters to their ones, our tests lie in the moderate

velocity range and low applied contact loads, good degree of agreement between our results and their results in that there is a threshold velocity before which the wear rate is decreased with increasing sliding speed (2.36 m/s in our case), and beyond which, an increase in the wear rate is observed with increasing the sliding velocity. This is attributed to localized surface thermal softening.

As contact load is increased the lateral cracks and hence brittle fracture becomes the dominant factor over plastic deformation and hence the speed value has no longer any effect.

In the MMC specimens it seems that the load range [2.5-7.5] N. is a stable range in which the bonding between Alumina particles and the Aluminum matrix is enhanced, this enhancement is obviously seen as the load is increased within this range in the high velocity region where plastic deformation is dominant over brittle fracture.

### Micro Roughness VS. Sliding speed And Spring load

Figure(5.31) shows the roughness (Ra) of worn surfaces as a function of load and speed, It is clear that Ra increases with both load and sliding speed, and it is also clear that the Ra's for the [2.5 to 7.5] N. load range are comparable which supports the previous discussion of wear results.

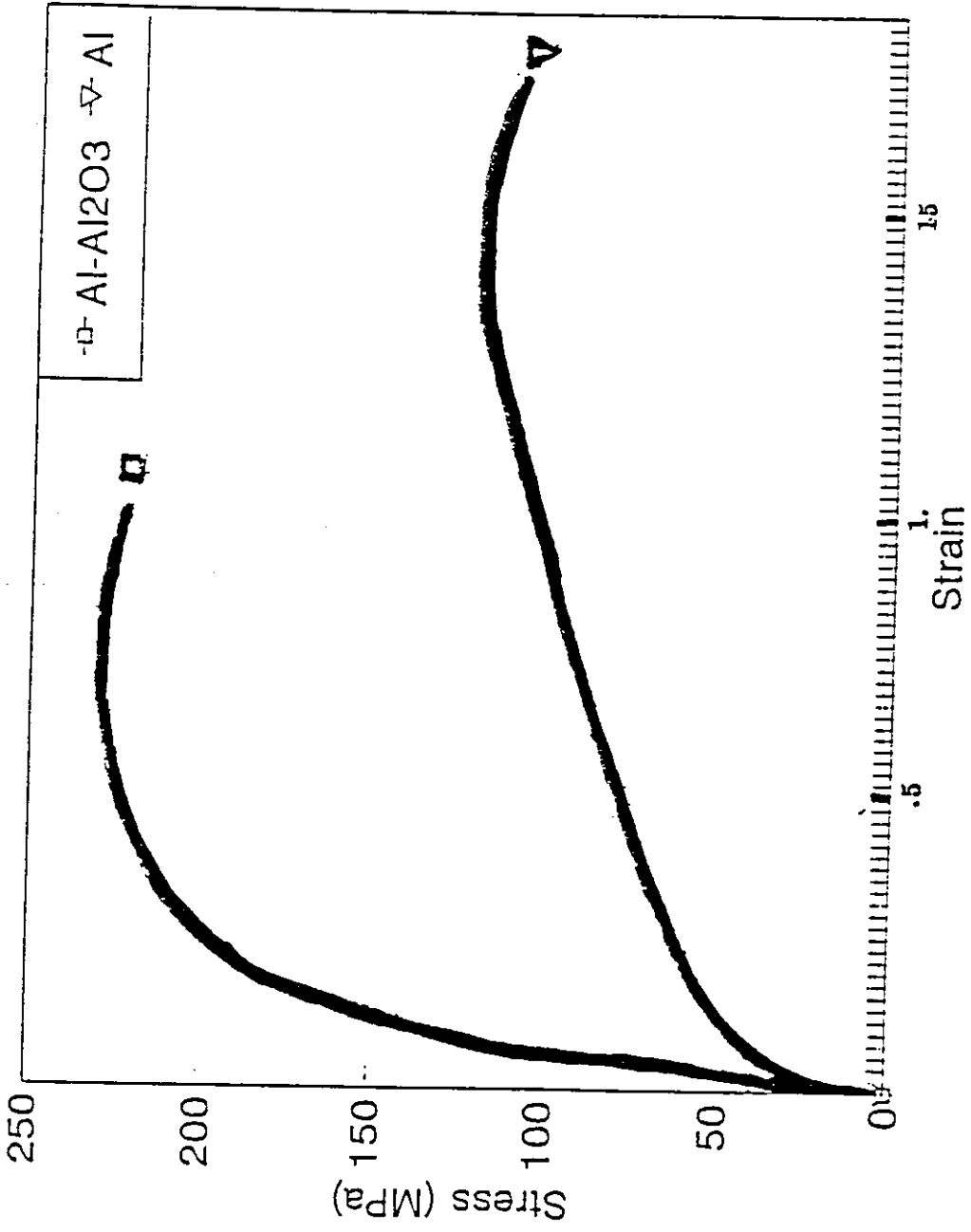


Figure 5.18: True Stress True Strain curve

Figure (5.18) True Stress strain curve



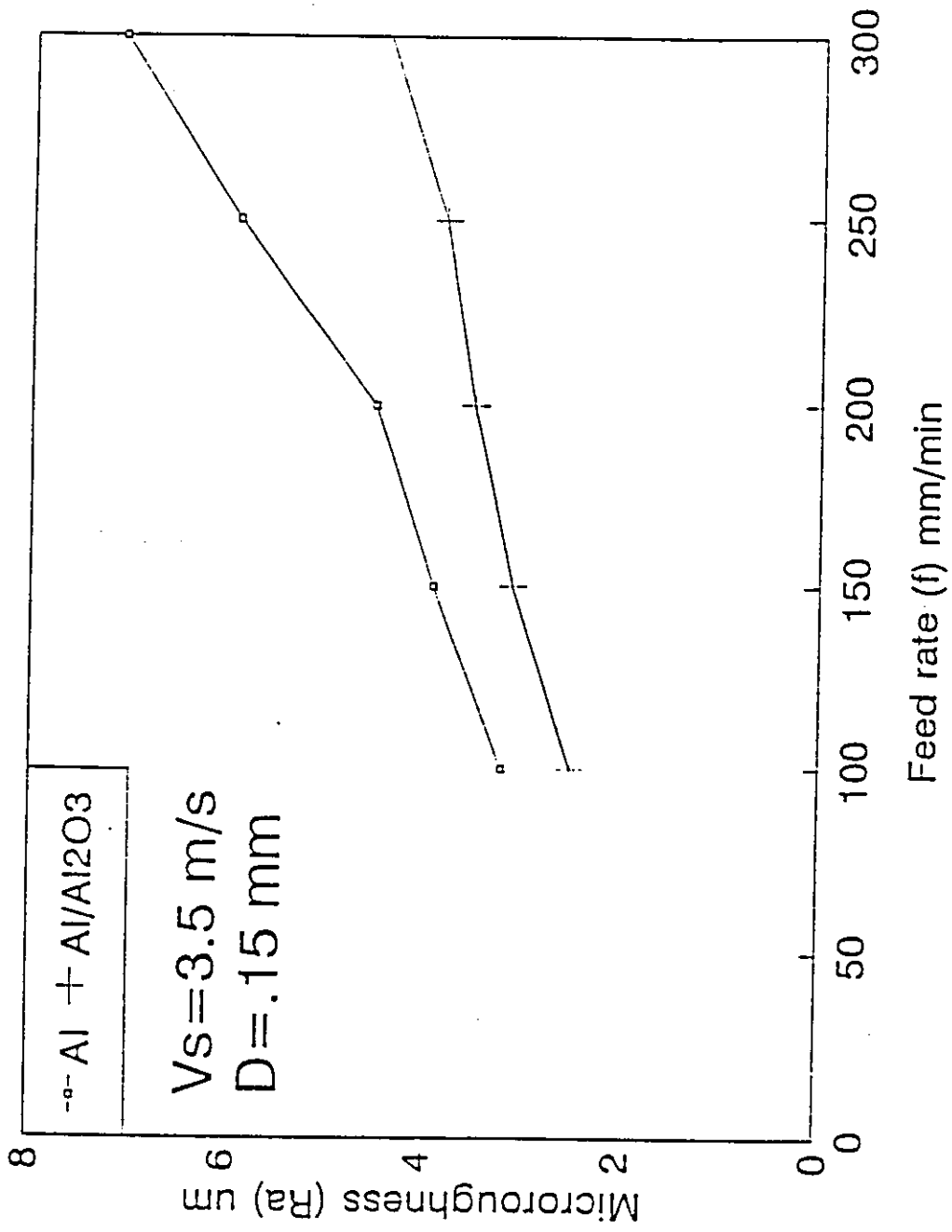


Figure 5.19: Ra Vs. Feed Rate

Figure (5.19) Microroughness Vs. Feed Rate

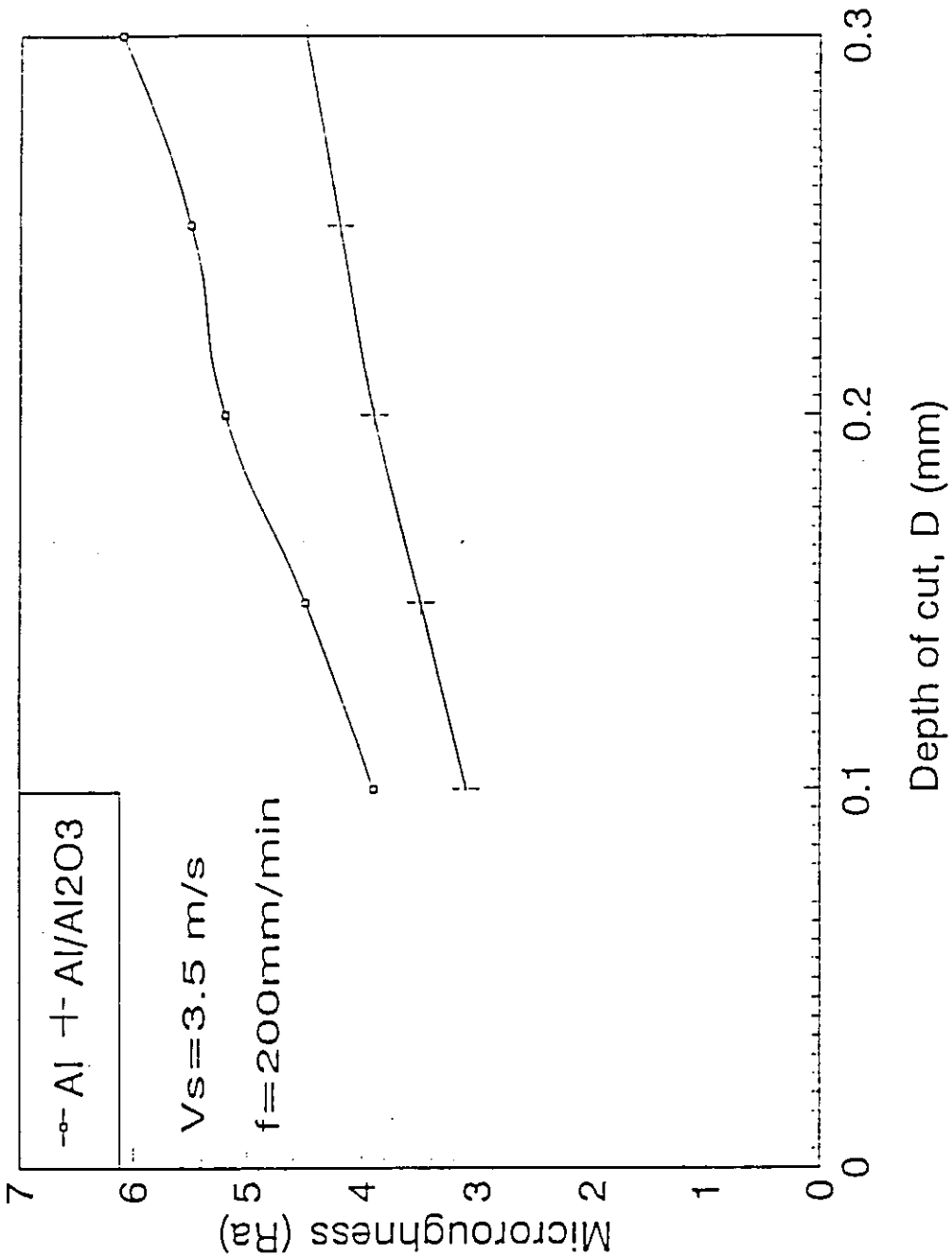


Figure 5.20: Ra Vs.Depth of Cut

Figure (5.20) Microroughness Vs. depth of cut

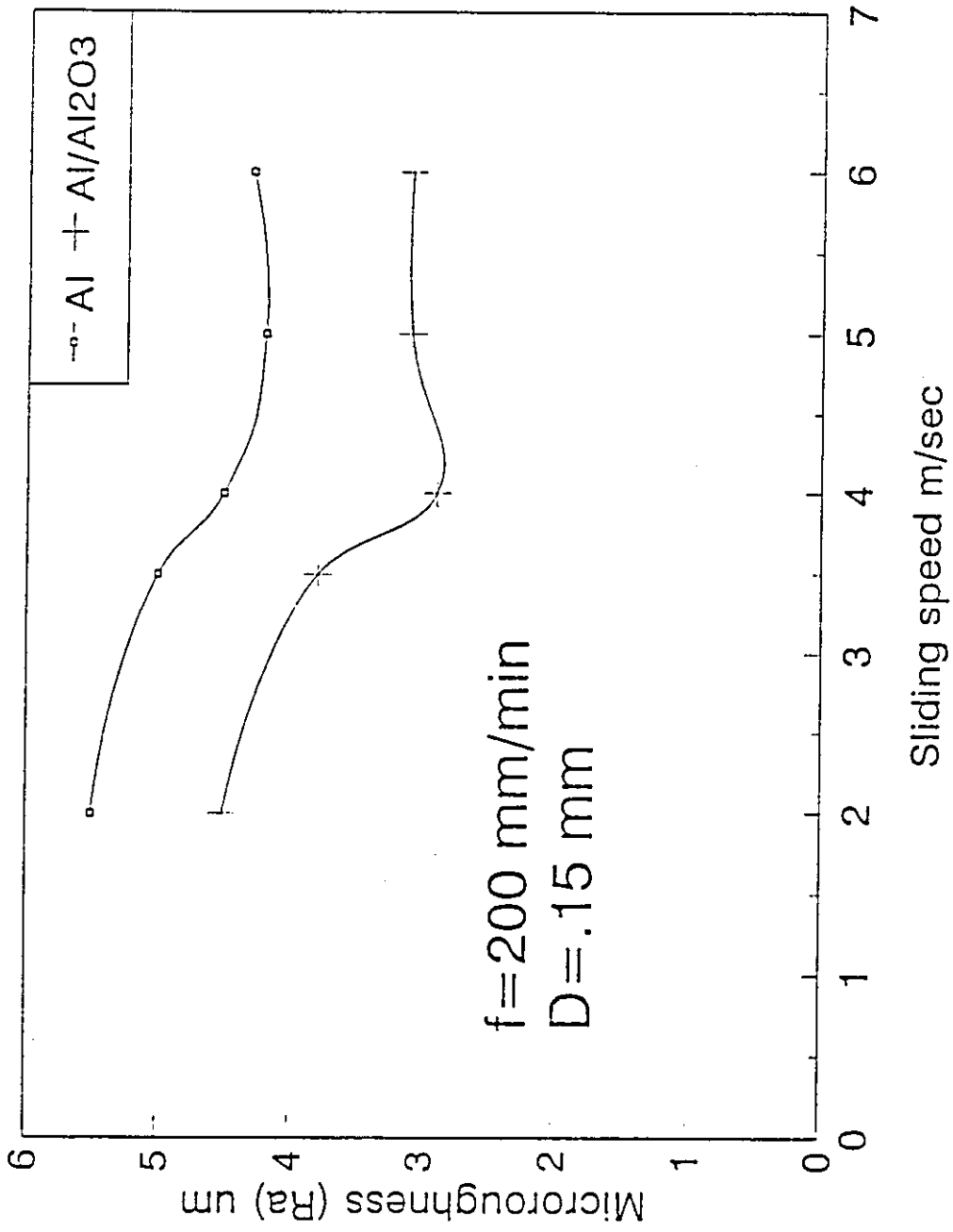


Figure 5.21: Ra Vs. Sliding speed

Figure (5.21) Microroughness Vs. Sliding Speed

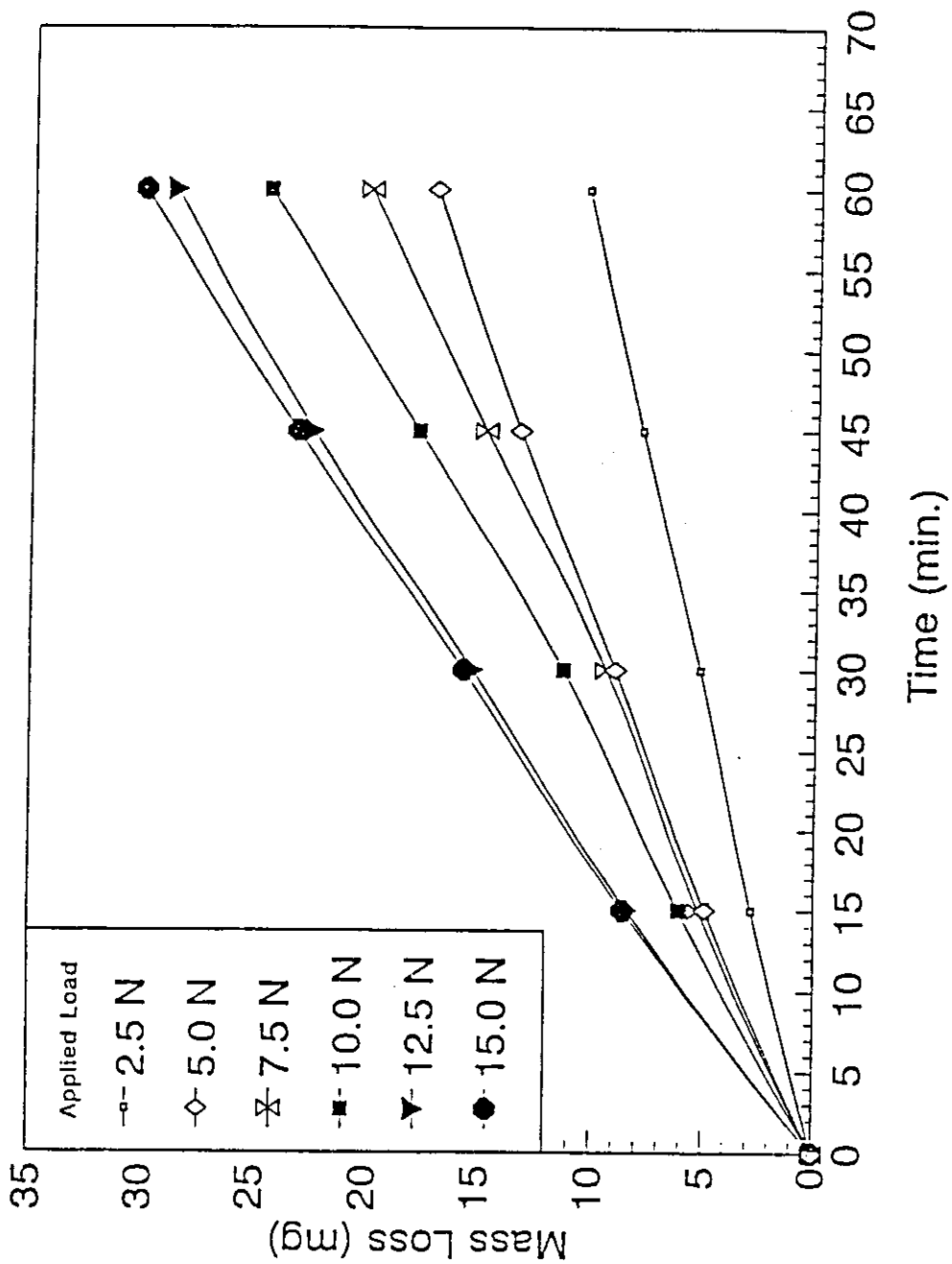


Figure 5.22: Mass Loss with Time

Fig (5.22) Mass Loss With Time.  
Sliding Speed 1.57 m/sec

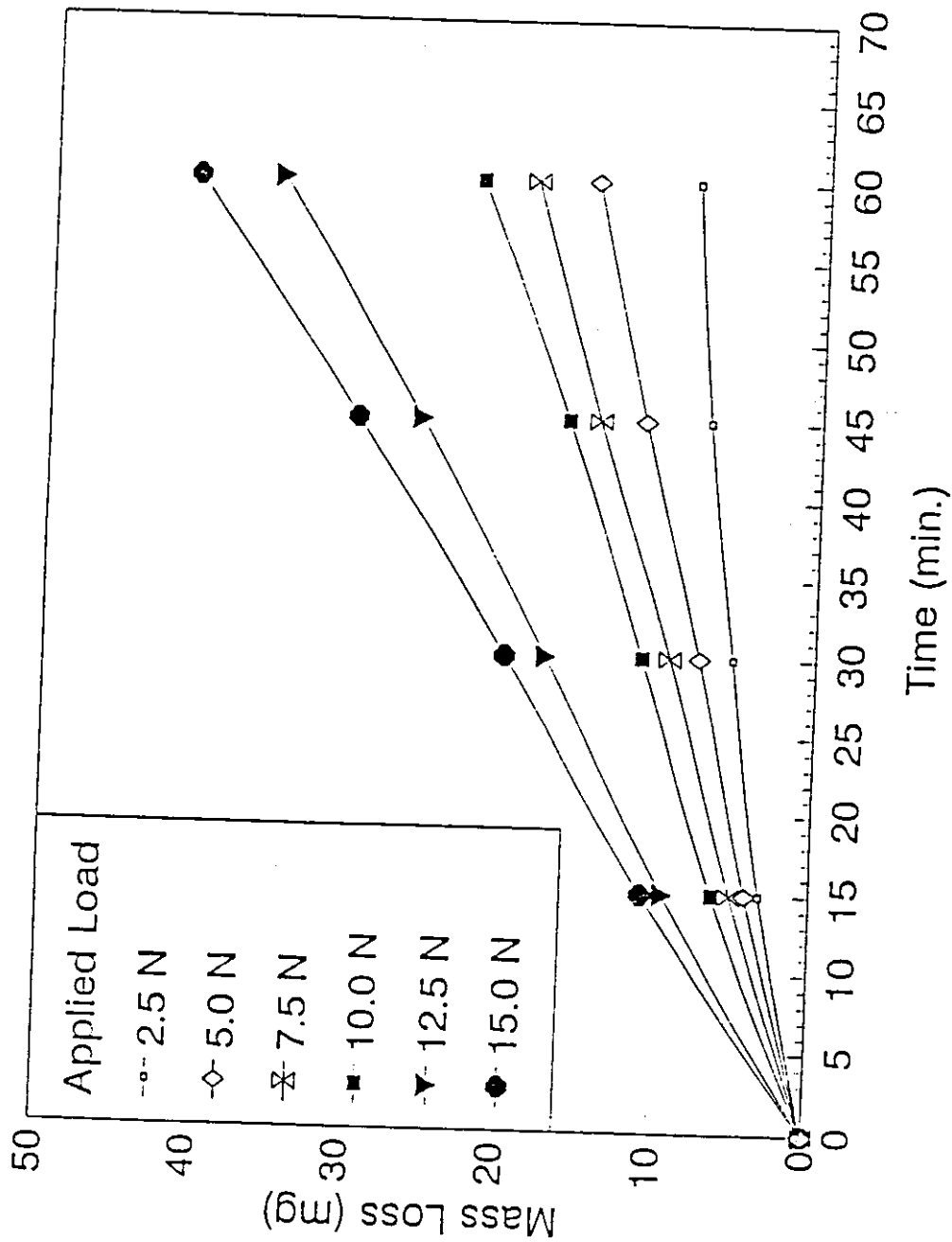


Figure 5.23: Mass Loss with Time

Fig (5.23) Mass Loss With Time.  
Sliding Speed 1.96 m/sec

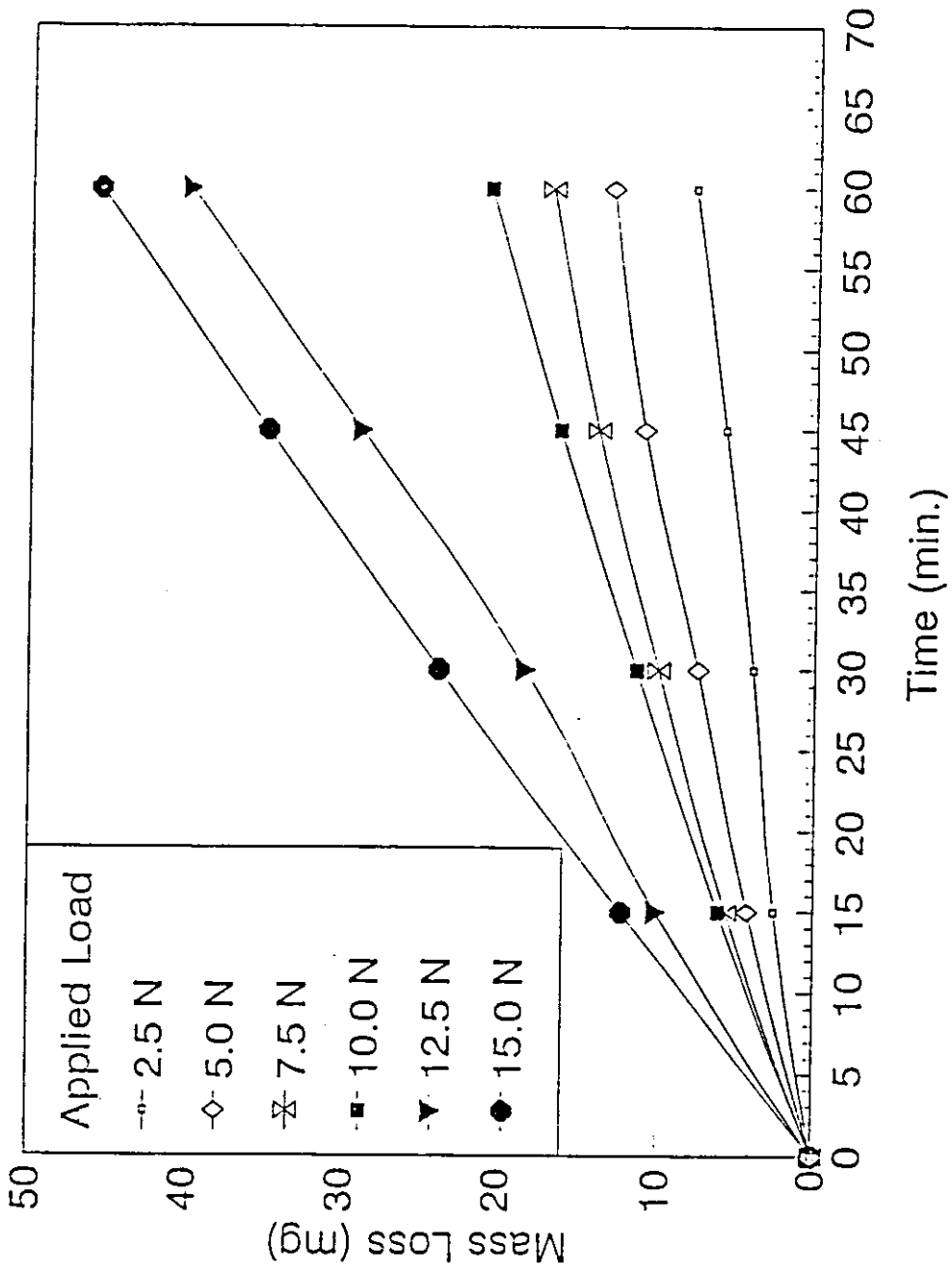


Figure 5.24: Mass Loss with Time

Fig (5.24) Mass Loss With Time.  
Sliding Speed 2.36 m/sec

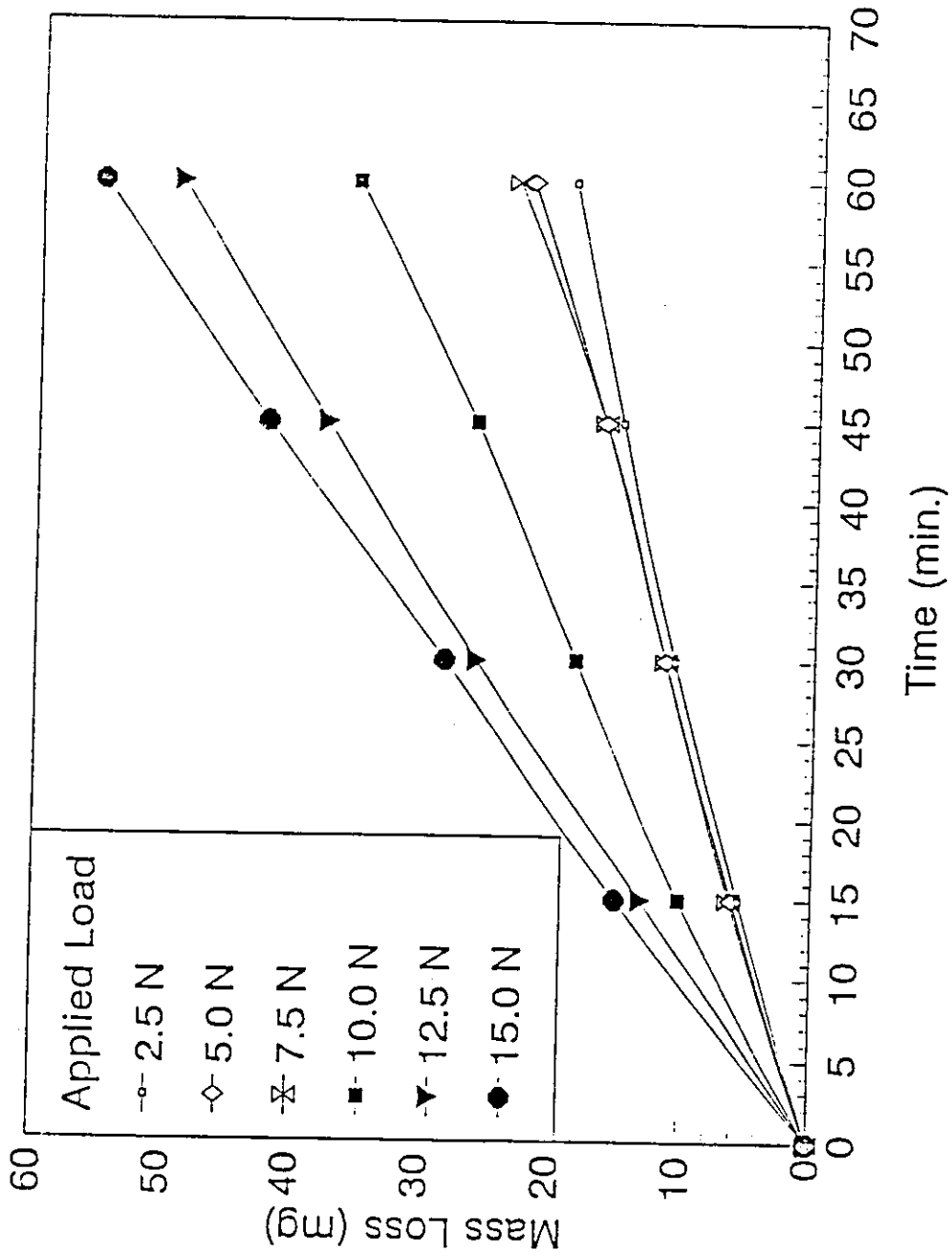


Figure 5.25: Mass Loss with Time

Fig (5.25) Mass Loss With Time.  
Sliding Speed 2.75 m/sec

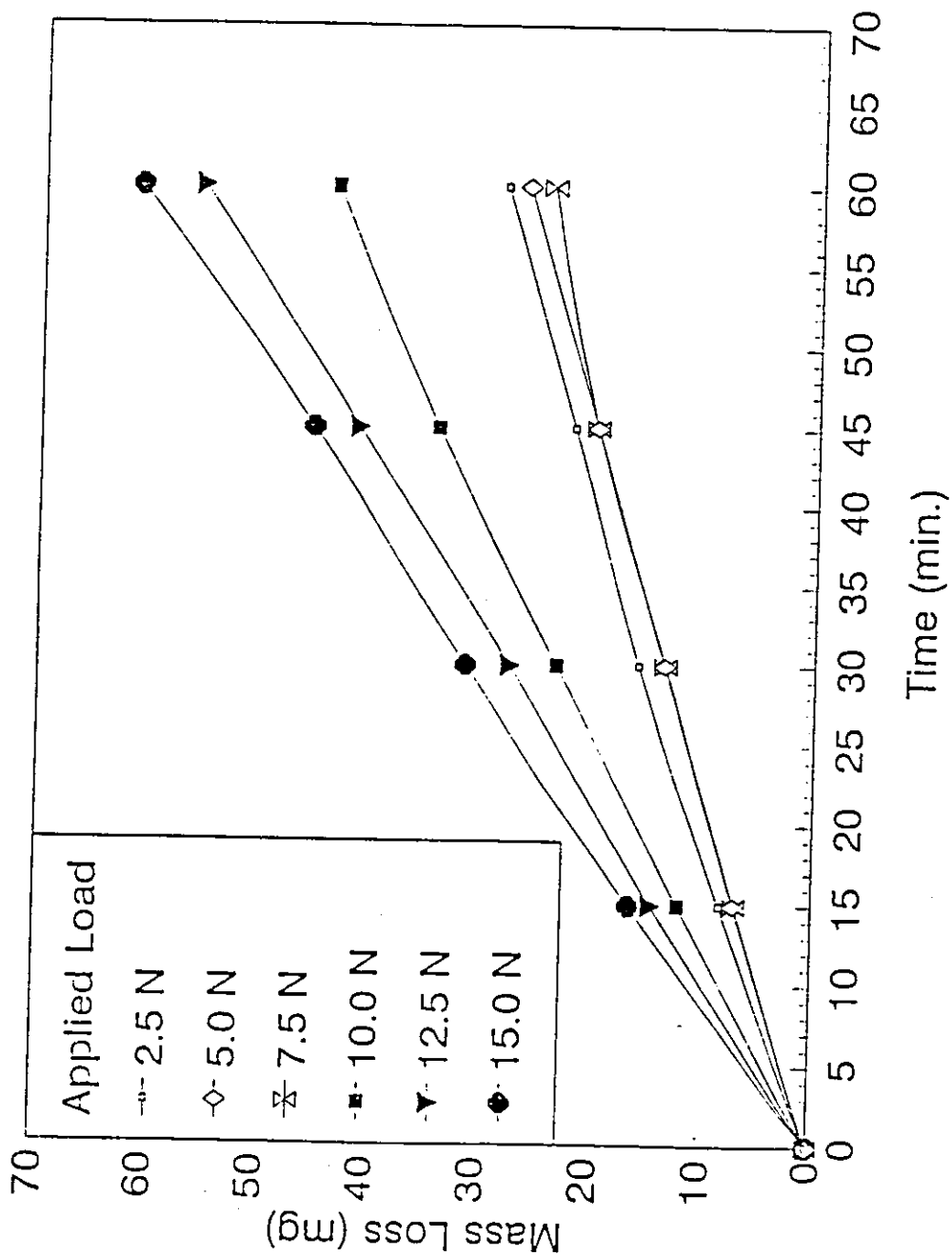


Figure 5.26: Mass Loss with Time

Fig (5.26) Mass Loss With Time.  
Sliding Speed 3.14 m/sec



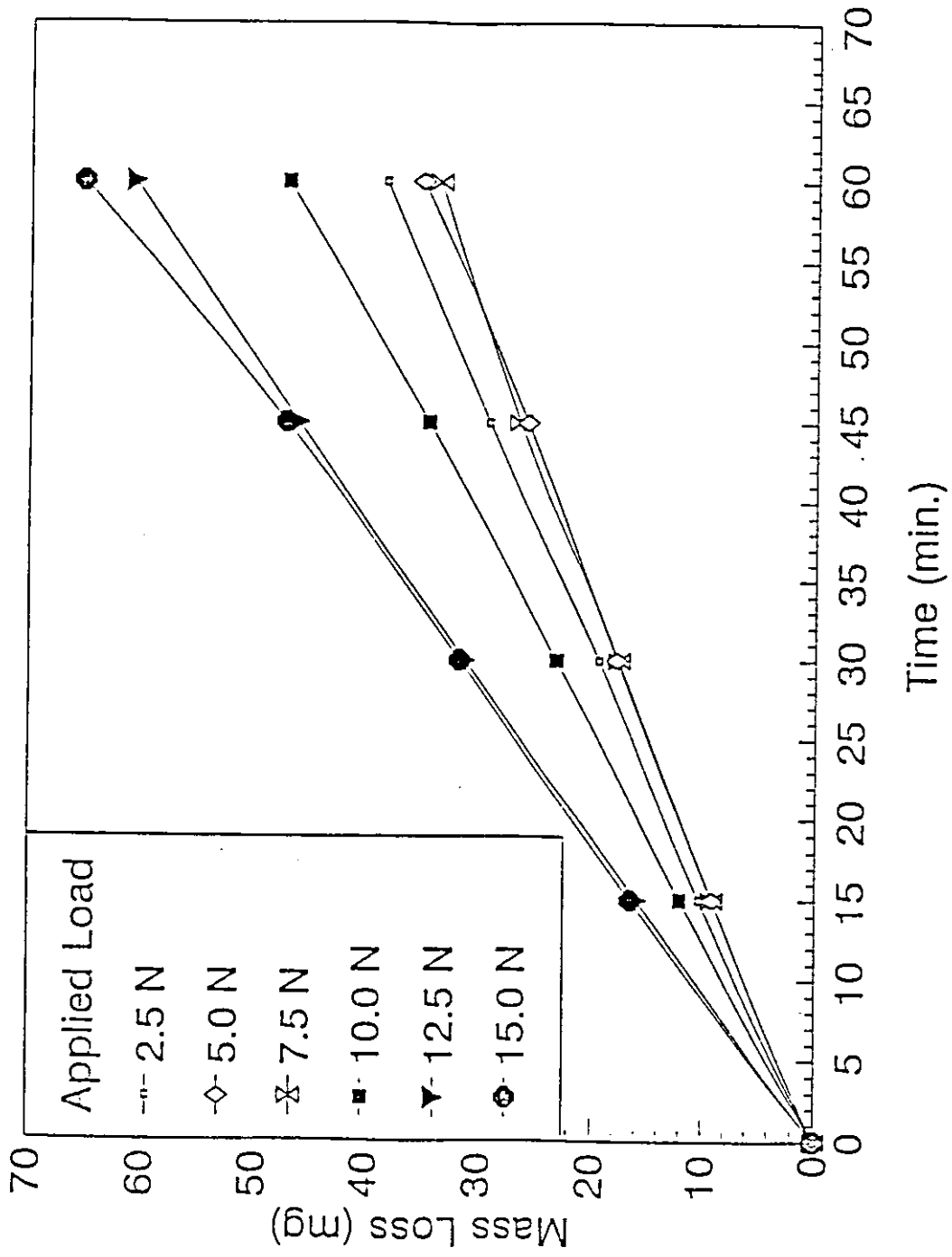


Figure 5.27: Mass Loss with Time

Fig (5.27) Mass Loss With Time.  
For Speed 3.53 m/sec

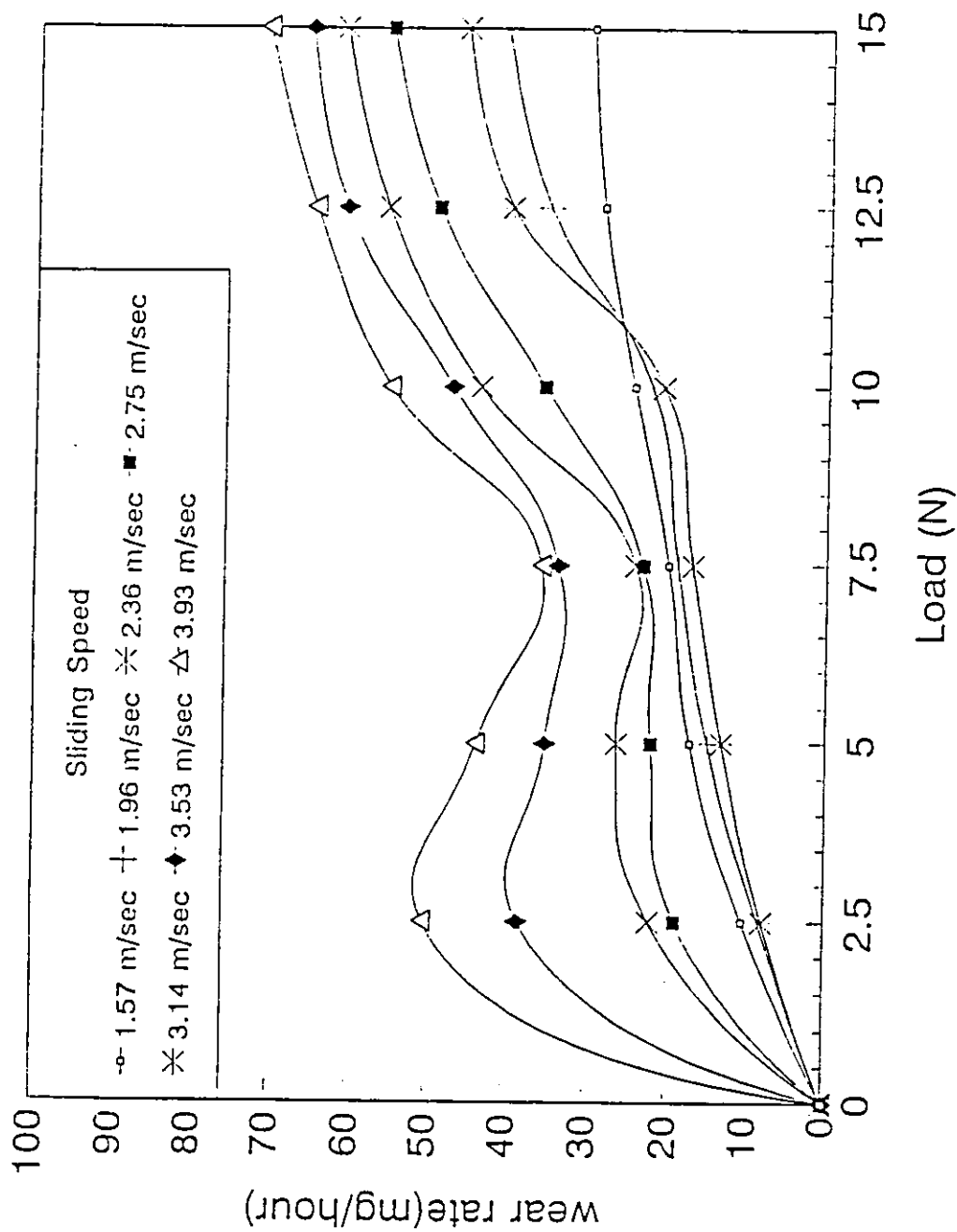


Figure 5.29: Wear Rate with load

Figure (5.29) Wear Rate With Applied Load

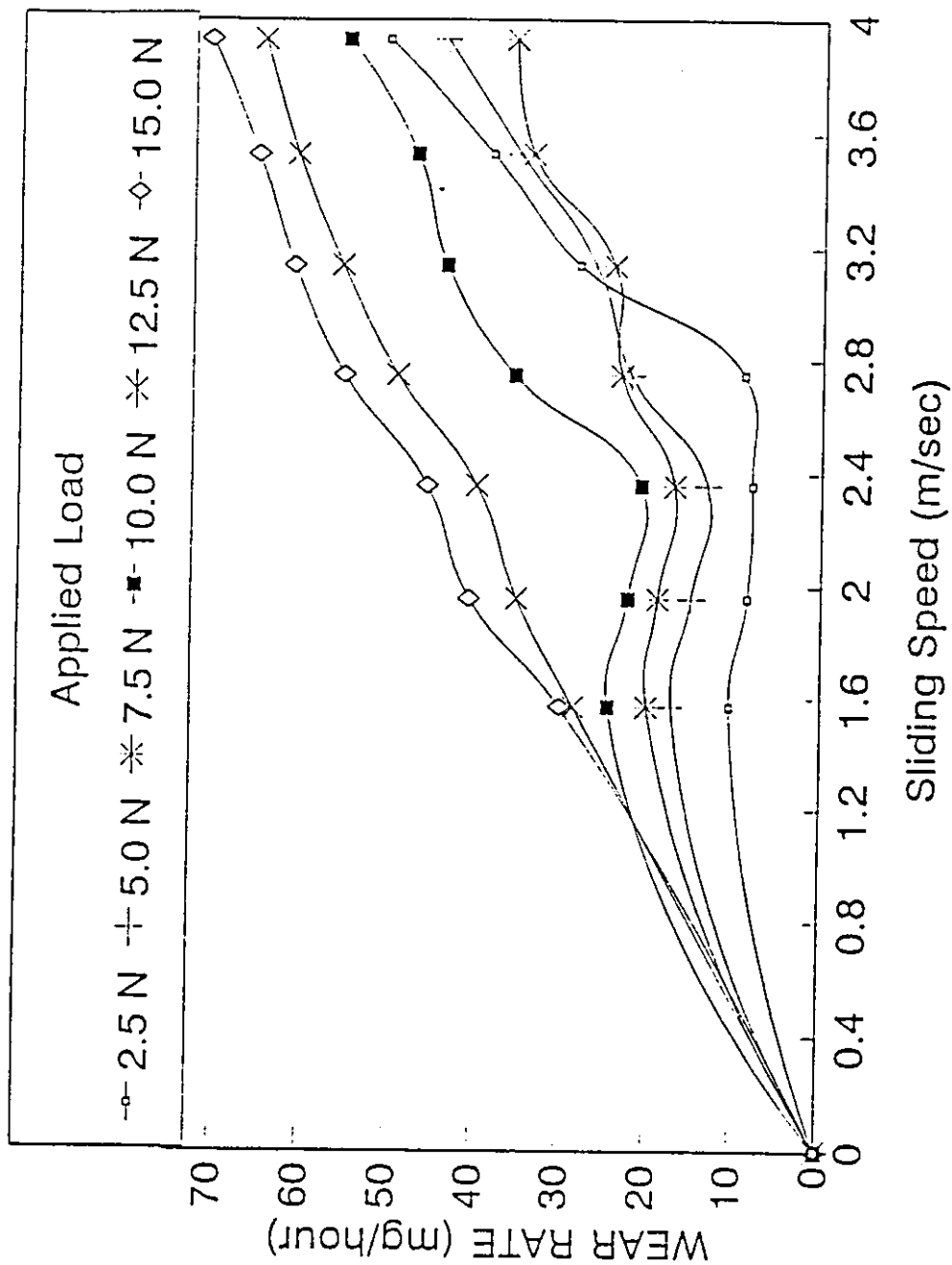


Figure 5.30: Wear Rate with sliding speed

Figure (5.30) Wear rate with Sliding Speed

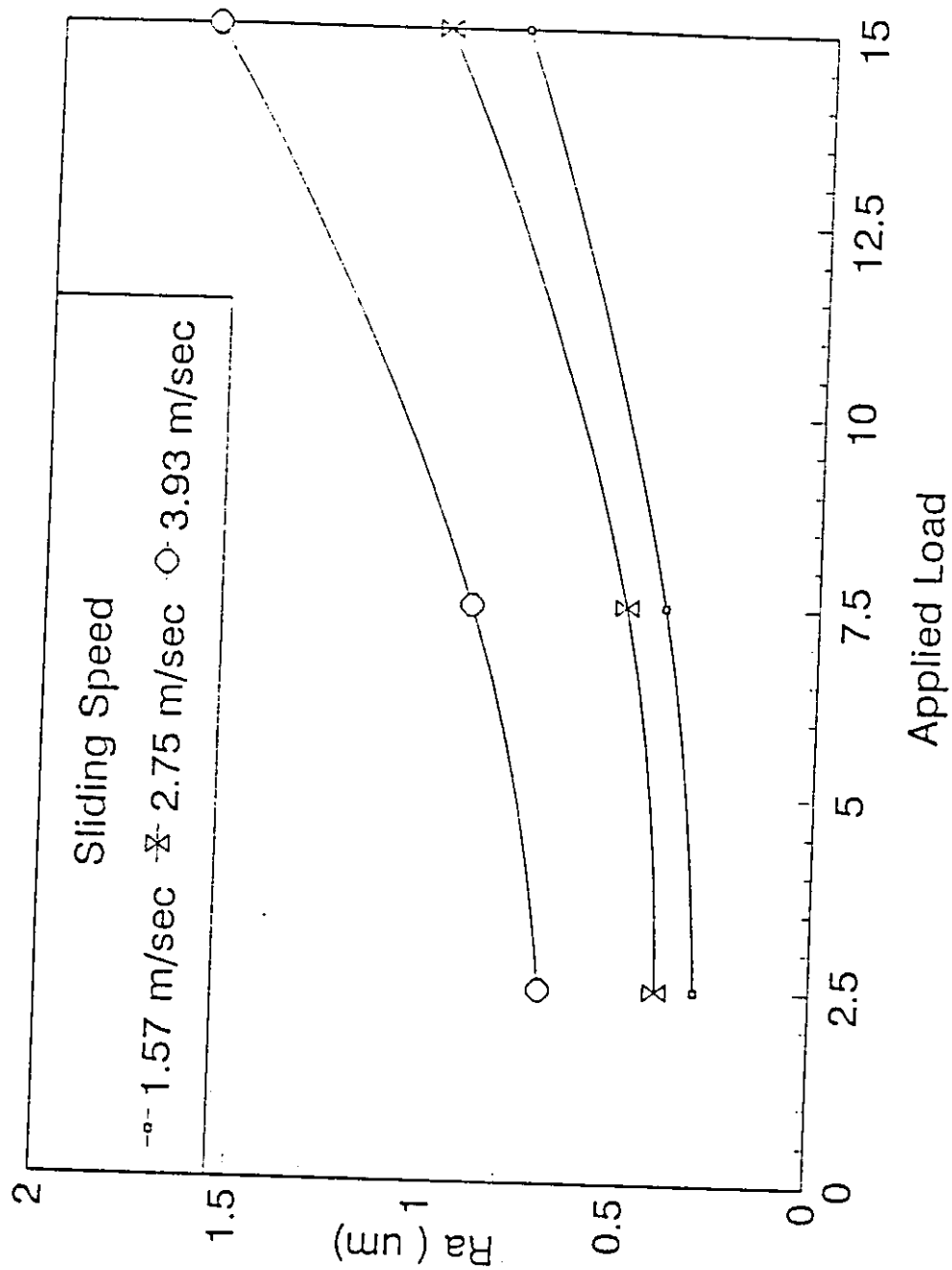


Figure 5.31: roughness with Load and Sliding speed

Figure (5.31) Microroughness Test (Ra) For Selected Worn Surfaces

## Chapter 6

# Conclusions and Recommendations for Future Work

### 6.1 Conclusions

From the results obtained throughout this work, the following conclusions are drawn:

1. Fabrication of  $Al_2O_3$  metal matrix particulate composite was successfully produced using the developed modified vortex technique, in which the vortex parameters are optimized.
2. The produced composite material was found to be completely sound, minimum microsegregation and with high ceramic volume fractions.
3. Mechanical properties tests showed that an increase in strength is achieved by adding  $Al_2O_3$  particles to pure Aluminum, and the ultimate strength is doubled, i.e. increased from 120 Mpa to 240 Mpa. Furthermore, Hardness tests revealed an increase of BHN from 55 for pure Al to 80 for the composite material.
4. The fabricated composite material has indicated better machinability characteristics than pure aluminium within the range of cutting conditions investigated throughout this work.

3. Similarly the fabricated composite material indicated better wear characteristics than pure aluminium, also under low and moderate loads the wear rate is found to be comparable with carbon steel. However under high loads, carbon steel is superior to the composite in wear resistance.

## 6.2 Recommendations

The following points are worthwhile to be investigated and suggested for future work:

1. Investigation of adding Mn as a reactive agent to pure aluminum and its effect on ceramic particles wettability.
2. preparation of the spinel  $[MgO, Al_2O_3]$  and adding it to the molten Aluminum, and study its effect.
3. Investigation of Alumina content on the metal matrix strength.
4. Investigation of the dynamic strength of the AL- $Al_2O_3$  metal matrix composites.
5. Investigation of thermal and electrical Conductivity of the AL- $Al_2O_3$  metal matrix composites.
6. Investigation of the temperature effect on the hardness of the composite material.
7. Preparation of the AL- $Al_2O_3$  metal matrix composites in micro-gravity fields.

# Bibliography

- [1] Pardeep Rohatgi  
"Cast metal matrix composites.", Metal hand book ASM, vol.15,  
840-854, 1986.
- [2] P. Schiller "Advances in composite materials",  
Advances in composite materials, vol.2 , 1-3, 1978.
- [3] S. V. Prasad and P. K. Rohatgi" Tribological properties of Al alloy  
particle composites",Journal of metals , 22-26, 1987.
- [4] , M. K. Surapa and P. K. Rohatgi, "Preparation and properties of  
cast AL - Ceramic particle composites ",Journal of Material Science,  
Vol. 16, 983-993, 1981.
- [5] H. A. Katman " Carbon Reinforced metal matrix composites",  
Us. Patent 4.376,808, 1983.
- [6] A. Banerjee; P. K. Rohtagi and W. Reil,"Role of wettability in  
the preparation of metal matrix composites", Metallography, vol.38,  
356- 362 , 1989.
- [7] M. K. Surapa and ; P. K. Rohtagi," Production of Aluminum -  
Graphite particle composites using copper coated graphite parti-  
cles", Met. Technical., vol.5, 358-363, 1978.
- [8] F. A. Badia, American Foundry men's Society, Vol. 79, 347-350,  
1971.



- [9] A. J. Mcevoy; R. H. Williams and I. G. Higgin Botham, " Metal-non-metal interfaces- wetting of magnesium oxides by Al and other metals ", Journal of material Science, vol.11, 297-..., 1976.
- [10] Nawal Izzet Abdul Lattef and Abdul Razaq Ismail Khader, " Preparation of Al- $Al_2O_3$ -MgO cast particulate composites using MgO coating technique ",Journal of Material Science, Vol. 4, 385-388 , 1985.
- [11] Yang Jingyu and D. D. L. Chung, " Wear of bauxite-particle reinforced Al-Alloys",Wear, Vol. 135, 53-65, 1989.
- [12] A. Melander; M. Rolfson ; S. Savage and E. Larsson, " Behaviour of short fatigue cracks in a short alumina fiber-reinforced AL-2Mg-alloy",Scandinavian Journal of Metallurgy, Vol.21 , 2- 10, 1992.
- [13] P. K. Ghosh and S. Ray ," Effect of mixing parameters on the microstructure of compocast Al(Mg)- $Al_2O_3$  particulate Composite ",Zeitschrift Fur MetalKunde,Vol. 9, 53-59, 1989.
- [14] P. k. Ghosh and S. Ray, "Effect of porosity and alumina content on the mechanical properties of compocast AL alloy - $Al_2O_3$  particulate composite ",Journal of Material Science Vol. 21, , 1667-1674, 1986.
- [15] G. R. Cappleman; J. F. Watts and T. W. Clyne " The interface region in squeeze infiltration composites containing S-alumina fiber in an Al matrix",Journal of Material Science, Vol. 20, 2159-2168, 1985.
- [16] P. K. Ghosh; and S. Ray," Influence of holding temperature and stirring speed on the surface reaction layer on - $Al_2O_3$  particles embedded in compocast Al-Mg alloy",Zeitschrift fur MetallKunde, Vol. 81, 525-529, 1990.

- [17] R. Mehrabian, " A fundamental study of a new fabrication technique for fiber reinforced Al matrix composites", Report on Us. army research office contract Dagg 29-76-G0170, April, 1980.
- [18] A. K. Dhingra and W. K. Kreuger "Fiber FP contain Yarn" Report by E.I. Dupont de Nemours and Co.,wilmington , Dl. 1974 .
- [19] S. V. Pepper," Shear-strength of metal sapphire contacts", J. appl. phys. , vol.47 , 801-807, 1976.
- [20] A. Munitz; M. Metzger and R. Mehrabian" Interface phase in Al-Mg-Al<sub>2</sub>O<sub>3</sub> composites", MET. Trans., vol.10a, 1941-1949, 1979.
- [21] A. Mnn, MET., vol.410a, 1941-1950, 1979.
- [22] H. C. Lee and M. S. Kim , " Fabrication methods of Al-short fiber alumina metal matrix composites processed by compocastings", Trans. Tech. Publ., Vol. 51, 471-475, 1991.
- [23] Kiuchi; Manabu; Takagi; Shigeyuoshi; Sugyama and Sumio, " Al-based particle reinforced composite sheets made by complex mashy- state processings and their formability ", Sumitomo light metal technical reports , Vol. 31, 38-45, 1990.
- [24] T. W. Clyne; M. G. Bader; G. R. Cappleman and P. A. Hulbert, " The use of S-alumina fiber for metal matrix composites" Journal of Material Science, Vol. 20, 85-96, 1985.
- [25] A. Mortensen and J. A. Cornie," Fundamental aspects of solidification processing of metal matrix composites by pressure infiltration techniques",Israel Journal of Technology, Vol. 24, 359-367, 1988.

- [26] A. Mortensen; L. J. Masur; J. A. Cornie and M. C. Flemings, "infiltration of fibrous preforms by a pure metal part I. Theory", Metallurgical Transactions, Vol. 20a, 2535-2549, 1989.
- [27] A. Mortensen; L. J. Masur; J. A. Cornie and M. C. Flemings, "Infiltration of fibrous preforms by a pure metal part II. Experiment", Metallurgical Transactions, Vol. 20a, 2549-2557, 1989.
- [28] V. Michaud and A. Mortensen, "Infiltration of fiber preforms by a binary alloy", Metallurgical Transactions, Vol. 23a, 2263-2280, 1992.
- [29] P. Jarry; V. J. Michaud; A. Mortensen; A. Dubus; R. Collet and Terard, "Infiltration of fiber preforms by an alloy, die casting", Metallurgical Transactions, Vol. 23a, 2281-2289, 1992.
- [30] T. Lim; Y. H. Kim ; C. S. Lee and K. S. Han, "Fabrication and mechanical properties of Al metal matrix composite materials", Journal of Composite Materials, Vol. 26, 1062- 1086, 1992.
- [31] A. Wang and I. M. Hutchings , " Wear of alumina fiber - Al metal matrix composites by two body abrasion " Material Science and Technology , vol.5, 71-76, 1989. **436763**
- [32] W. J. Clegg; I. Horsfall; J. F. Mason and L. Edwards, " Tensile deformation and fracture of AL- Saffil metal matrix composites " Acta Metallurgica, vol.36, 2151-2159, 1988.
- [33] M. Manohran and J. J. Lewandoveski " In - Situ deformation studies of an Al metal matrix composite in a scanning electromicroscope", Scripta Metallurgica , vol.23, 1801-1804, 1989.

بعد تصنيع متراكبات الالومنيوم بمواصفات معدنية جيدة تمنا بإجراء بعض الفحوص للكشف عن خواص المنتج الميكانيكية حيث أظهر فحص الضغط *Compression Test* مقاومة أكبر للمنتج بإضافة الحبيبات *Al2 O3* من مقاومة الالومنيوم الصافي، حيث تضاعفت مقاومة متراكبات الالومنيوم للاجهاد الأقصى بمقدار الضعف. وأظهر فحص الصلابة *Hardness Test* تحسناً كبيراً في مقاومة سطح المعدن.

أظهر فحص نعومة سطح المعدن *Roughness Test* أنه بإضافة حبيبات أكسيد الالومينوم *Al2 O3* إلى الالومنيوم النقي فإن سطح المنتج يكون ذا نعومة أكثر عند جميع ظروف القطع من نعومة سطح الالومينوم النقي.

وأخيراً أظهر فحص الإهتراء *Wear Test* أن متراكبات الالومنيوم المدعمة بحبيبات أكسيد الالومينوم *Al2 O3* تقاوم الإهتراء عند ظروف التحميل المنخفض والمتوسط بنفس كفاءة مقاومة بعض أنواع الفولاذ، غير أن الفولاذ يكون أكثر مقاومة للهريان في ظروف التحميل العالية.