ACTIVATED SINTERING OF ALUMINIUM-COPPER POWDER-METALLURGICAL PARTS

A Dissertation
submitted in partial fulfilment of the
requirements for the award of the Degree
of
MASTER OF ENGINEERING
in
PHYSICAL METALLURGY

By

PRIYA VRAT THAREJA

DEPARTMENT OF METALLURGICAL ENGINEERING
UNIVERSITY OF ROORKEE
ROORKEE (INDIA)
August 1974

UNIVERSITY OF ROORKEE
ROORKEE

CERTIFICATE

I hereby recommend that the thesis being submitted by Shri P.V. Thareja entitled 'Activated Sintering of Aluminium-Copper Powder Metallurgical Parts' be accepted as fulfilling this part of requirement for the degree of Master of Engineering in Metallurgical Engineering (Physical Metallurgy);

It is a record of his own work carried out by him under my supervision and guidance from January 1 to August 24, 1974.

The matter embodied in this dissertation has not been submitted for the award of any other degree.

(G.S. UPADHYAYA)
ASSOCIATE PROFESSOR
SUPERVISOR

Department of Metallurgy
University of Roorkee
Roorkee (UP)

27/8/74
Dated:

ABSTRACT

The present investigation forms a part of long term studies carried out in the department on activated sintering of Aluminium alloys. Al-3%, 4%, 5% Cu alloy were activated sintered with addition of Boron isothermally at 600°C for different periods in vacuum. In addition the effect of compacting pressure on sintering behaviour of above alloys were also carried out and the optimum compacting pressure was selected for this work on isothermal sintering.

It was observed that Boron activates sintering of the compacts such that with increase in Copper content the densification parameter and hardness peaks, which is observed during isothermal sintering, increase in magnitude as well as its position shifts to the lesser sintering periods. The results have been interpretted on the basis of different extent of liquid phase formed during sintering and its possible effects on the sintering mechanism.

ACKNOWLEDGEMENTS

The author is highly grateful to Dr. G.S. Upadhyaya, Associate Professor, Department of Metallurgical Engineering, University of Roorkee, for suggesting the problem, inspiring guidance and invaluable discussions throughout the course of this work.

Thanks are due to Sri P.S. Misra, for his guidance, keen interest and invaluable suggestions in the preparation of this dissertation.

The author takes the opportunity to thank Dr. M.N. Saxena, Professor & Head, Department of Metallurgical Engineering, University of Roorkee, Roorkee, for providing with the necessary facilities in the department.

The author also thanks the technical staff of Metallurgical Engineering Department for their help and cooperation.

Pv Thareja

(PRIYA VRAT THAREJA)

CONTENTS

CERTIFICATE i

ABSTRACT ii

ACKNOWLEDGEMENT iii

CONTENTS .. .. .. iv - vi

CHAPTER TITLE PAGE

I INTRODUCTION 1

II LITERATURE REVIEW 4

II.1 Introduction 4

II.2 Sintering of Al metal Powders 5

II.3 Sintering of Al-Cu Alloys 8

II.3.1 Binary Alloys of Al-Cu 8

II.3.2 Duralumin Type Alloys of Cu 17

II.4 Activated Sintering of Al and its alloys 26

II.5 Scope of the Present Work 30

III EXPERIMENTAL PROCEDURE 32

III.1 Powder Characteristics .. 32

III.1.1 Aluminium Powders .. 32

III.1.2 Copper Powders 32

III.1.3 Boron Powder 33

III.2 Blendhing 33

CHAPTER TITLE PAGE

III.3 Green Compact Formation .. 34

III.4 Sintering of Compacts 34

III.5 Density Measurement 35

III.5.1 Densification Parameter .. 36

III.6 Hardness measurement .. 36

III.7 Microstructural Studies .. 36

IV RESULTS 37

IV.1 Effect of Compacting Pressure on Properties of Al-Cu Alloys 37

IV.1.1 Green Density 37

IV.1.2 Densification Parameter .. 37

IV.1.3 Hardness 39

IV.2 Effect of Boron Addition on Al Alloy System 39

IV.2.1 Densification Parameter .. 42

IV.2.2 Hardness 43

IV.3 Microstructural Studies .. 45

IV.3.1 Effect of Compacting Pressure on Microstructures of Al Alloys 45

IV.3.2 Effect of Sintering Period on Microstructures of Al Alloys 45

V DISCUSSIONS 48

V.1 Effect of Compacting Pressure on Sintering Behaviour of Al-Cu Alloys Compacts 48

V.2 Effect of Sintering Period on Properties of Al-Cu admixed powder Compacts 50

CHAPTER	TITLE		PAGE
	V.2.1 Aluminium Powder Compacts	..	50
	V.2.2 Al-3% Cu Alloys compacts	..	51
	V.2.3 Al-4% Cu Alloy compacts	..	52
	V.2.4 Al-5% Cu Alloy compacts	..	53
VI	CONCLUSIONS	..	57
	REFERENCES	..	59

CHAPTER I

INTRODUCTION

Technical feasibility for the powder metallurgical (P/M) parts fabrication process is derived from two physical effects, which are green 'densification or compressibility' and 'sintering'. Both effects are highly influential on properties and are related to each other through the properties chosen for use. Metal powders possessing the right combination of compressibility and sinterability for part fabrication are highly specialized and developed product. So these two important parameters are determined to a large degree by the dimensional and physical properties of individual particles. The second major physical effect fundamental to the P/M process is sintering, which is described as the bonding of adjacent surface of particles in a mass of metal powder or a compact by heating. Practically this process is carried out in controlled atmosphere/vacuum furnaces.

Sintering can proceed on the basis of solid-solid diffusion alone, or alternatively on the basis of mixed solid-solid and liquid-solid diffusion depending upon alloy systems and sintering temperature. Under either conditions the sintering process may be divided into three practically identifiable stages(1) as follows:

1. Stage 1 : Initial Bonding: comprises a conversion of mechanically interlocked particle surface with true grain boundaries approximately like those formed in castings.

2. Stage 2 : Neck growth: Neck growth comprises of massive diffu-

sion across newly formed grain boundaries and the distortion of particle shapes to form isolated pores in locations initially not mechanically interlocked.

3. Stage 3 : Pore Shrinkage : It comprises the migration of atom size vacancies away from isolated pore surface to the compact external surface.

These sintering stages are shown(1) in Fig.I.1. 80% of potential mechanical properties are obtaibed by sintering only enough to complete stage 2, and in some cases just enter stage 3 to the point of pore spherodization. This relates to the fact that dimensional precision and economic productivity both are adversely effected by prolonged stage 3 sintering. This practice is developed around sintering carried to the transition point between stages 2 and 3.

It is a matter of experimental observation that metal parts made by conventional pressing and sintering quite often i. takes an inordinately long time to achieve the desired density and strength, ii. lack in uniformity of strength along the dimensions of specimen, iii. lack in physical and mechanical properties such as density, elongation and impact resistances, iv. require high temperatures for sintering.

The process of activated sintering offers the possibility of overcoming these difficulties by modifying the metal powder surfaces. The application of an activation process rests upon the assumption that sintering is mainly a phenomenon connected with surfaces and therefore an activation of surfaces of powder

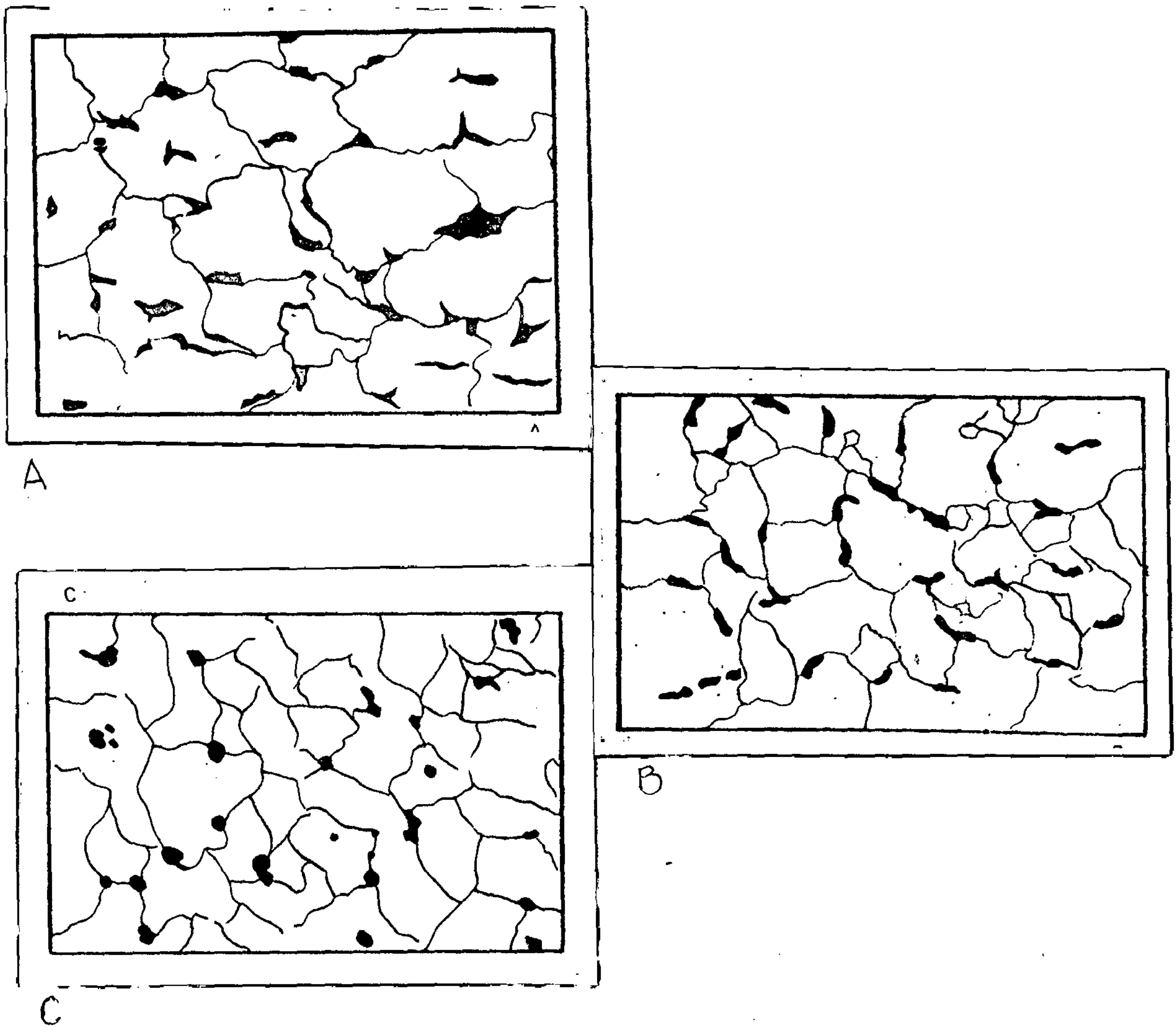


FIG.I.1 THREE STAGES OF SINTERING: A: STAGE-1 PARTICLE BONDS CHANGING FROM MECHANICAL TO METALLURGICAL VIA SOLID STATE DIFFUSION POROSITY COMPRISES THE ORIGINAL INTER PARTICLE SPACES; B: STAGE-2 MECHANICAL BOUNDARIES HAVE BECOME GRAIN BOUNDARIES AND SOME OF THESE HAVE BEEN RECRYSTALLIZED AND DISAPPEARED POROSITY HAS ISOLATED AND IS IN THE PROCESS OF SPHERODIZATION; C: STAGE-3 GENERAL RECRYSTALLIZATION IS COMPLETE AND POROSITY IS SPHERODIZED. SINTERING CONTINUES AT A SLOW RATE VIA VACANCY MIGRATION OF PORE SPACE TO SURFACE (1) must improve sintering. Free surface of a solid is contributed as a zone of discontinuity when the forces of attraction between atoms are no longer in equilibrium and lattice defects in the form of vacancies, foreign atoms and dislocations increase in number. These factors influence the structure of surface and the behaviour of the particles toward the surrounding medium.

The expression 'Activated Sintering' is used in connection with the sintering of powder compacts, where the rate of sintering is modified by some physical or chemical treatment of pow-der or compact by incorporating reactive gases e.g. H_2 moisture in HCl, NH_4F, NH_4Cl with a view to enhance densification, mechanical strength, magnetic and electrical properties. Several physical and chemical methods of activation are now available(19) but in the present investigation only one of the chemical method of activation is tried viz. sintering in the presence of very small quantities of alloying elements which have either mutual solubility or do not have solubility in the substrate.

CHAPTER II

LITERATURE REVIEW

II.1 Introduction:

Production of Al p/m parts is quite difficult, as it causes trouble in both of the critically import-ant production steps: compacting and sintering. In the compacting process Al powder has a tendency to cause galling and seizure of the tooling somewhat more readily than other powders. It has a tendancy to extrude between the moving and stationary parts of the die. It also has poor flow rates. And if the part is compacted successfully Al has a tendency to weld to the die, so that the part can not be ejected from the tooling. Even after these factors are completed successfully sintering remains another hurdle. It is difficult because of the presence of oxide films on Al powder particles. (Fig II.1) The scale of which is about 100 A^o thick and may be broken up by the Al powder itself making it possible for sintering to be achieved even by conventional methods.

Lubricants added to ease compaction of the powder cause sintering problems by generating decomposition products. Lubricants act as pore generators too. The residues left behind would interfere with the establishment of solid sinter bond and be the cause for inferior properties. Hence one of the suggested(2) lubricant is flake Al particles suspended in stearic acid dissolved in a commercial solvent.

The sintered parts are reported(3) to be distored and exhibit either macroporosity or property debilitating microscopic porosity, which they attribute to the expansion of air originally trapped during pressing operations.

Engineers have tried to overcome these difficulties, and have been successful in obtaining properties comparable to those obtained by wrought or cast alloys. Difficulties in compaction and ejection encountered in pure Al powders are lessened considerably by the addition of Cu(4), since Cu particles tend to dislodge adhesion of Al particles to die walls. The use of atomized alloy powder eliminates the molding and ejection difficulties entirely but the resulting products are generally of inferior quality because of greater rigidity of particles and therefore an increased porosity of the compact formed at conventional pressures of 30-50 tis (4.65 - 7.8 tonne/sq.cm.). On addition of particularly Cu to Al the powder on compaction (5) behaves as if it were in a semiplastic state, and compacts with great ease under the pressure applied ranging 3-20 tsi (.47 - 3 tonne/sq.cm) depending upon the desired density, the quantity of powder being pressed and the surface to transverse crosssectional area ratio. The high plasticity allowing high densification also produces good sintering characteristics because the elemental Cu powder additive diffuses into Al very rapidly and dimensional control is fairly easy to obtain.

II.2 Sintering of Al Metal Powder:

The credit points for successful Al p/m part production are the selection of right lubricants, die design and maintaining close control over the sintering process. The heat up rate during sintering is critical and must be kept above 20^{o}C/min. An angular taker of 10' on the die walls similar to the draft made on a casting mold helps prevent die sticking. The addition of copper (Cu) powder avoids other problems in the pressing of compact. The Cu powder coats the Al powder so that the die does not actually press Al and the compressibility is increased. In these alloys the high plasticity of soft Al powder allowed for unusual compactions$^{(4)}$ to be made in particular 3:1 :: l:d ratios, where diameter varied less than 1% over the entire length of 5" of the piece. The piece themselves could be pressed to density values as high as 97% theoretical.

Sintering of Al is usually conducted in Air, N_2, Helium or dissociated Ammonia at a temperature slightly below the fusion temperature. Cremer and Cardiano$^{(5)}$ worked on sintering of Al for 30 minutes at 620^{o}C and the results are plotted in Figs. II.2 and II.3. These figures compare favourably with wrought Al as far as strength characteristics are concerned, while the elongation falls somewhat short of normal. The effect of compacting pressure on Al compacts sintered in dissociated ammonia for one hour at 615^{o}C is given in Table I.

Aluminium attains complete density (2.7 g/cc) at already moderately low molding pressures because of its excellent plasticity. Bickedyke$^{(6)}$ obtained remarkable results with compacts hot pressed at 5 tsi (.78 tonne/sq.cm.) after initial cold compaction at 30 tsi (47 tonne/sq.cm.) reproduced in Table II.

FIG.II.1

A MICROPHOTOGRAPH OF A MICROSECTION THROUGH OXIDISED ALUMINIUM PARTICLES (X600) (2)

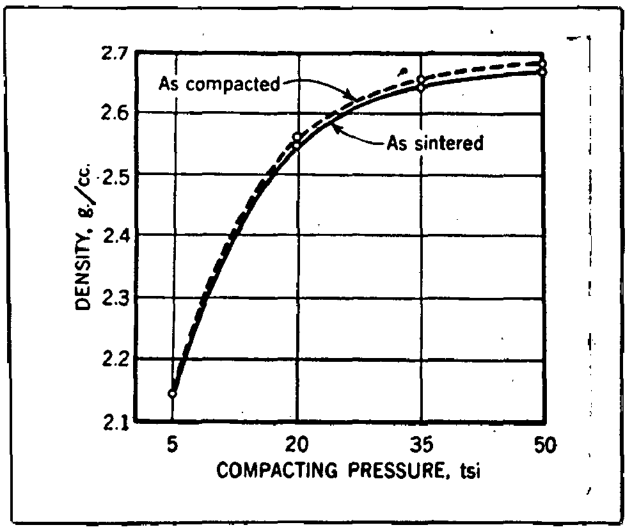


FIG.II.2

EFFECT OF COMPACTING PRESSURE ON THE DENSITY OF AIR SINTERED ALUMINIUM

(7)

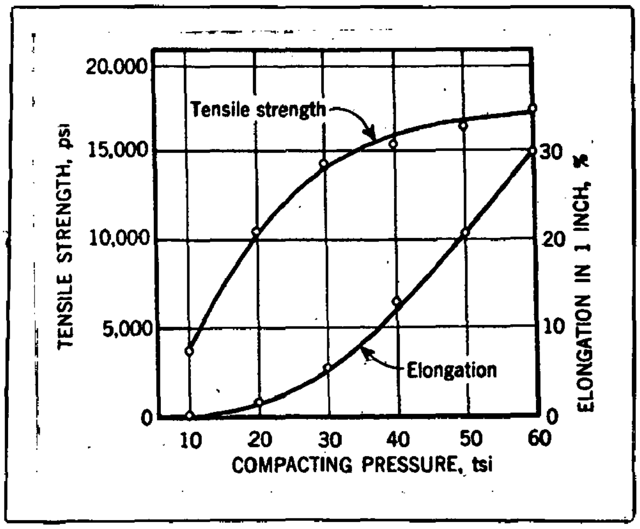


FIG.II.3

EFFECT OF COMPACTING PRESSURE ON TENSILE PROPERTIES OF AIR SINTERED ALUMINIUM (7)

TABLE I : EFFECT OF COMPACTING PRESSURE ON ALUMINIUM COMPACTS

Compacting Pr. tsi	Density g./cc	Yield Str. psi	Tensile Str. psi	Elong. in 1 in. %	Reduction of area %
10	2.44	6160	8900	4.7	3.1
20	2.60	6460	11810	46.8	63.7
30	2.69	6660	11850	48.4	59.4
40	2.69	7600	11780	37.5	47.7

TABLE II : MECHANICAL PEROPERTIES OF ALUMINIUM COMPACTS

Hot-Pressed at 5 TSI after Initial Cold-Compaction at 30 Tsi.

Pressing Temperature		Brinell hardness	Ultimate Strength psi	Elongation %
°C	°F			
300	570	33.7	12,600	1.0
400	750	30.6	12,400	2.0
500	930	28.4	12,200	4.5
600	1110	26.4	15,400	34.0

Effect of an increasing compacting pressure on density is pronounced only for low pressures. At the moderate pressure of 30 tsi (4.7 tonne/sq.cm.) nearly theoretical density values are obtained while higher pressures result only in minor increase in density. The physical properties closely follow the same rule.

II.3 Sintering of Al-Cu Alloys:

Sintered Al-Cu alloys are probably of greatest promise. These include binary alloys containing 1 - 10% Cu, and also duralumin type containing about 0.5% each of Mg, Mn in addition to 4%Cu.

II.3.1 Binary Alloys of Al-Cu:

Addition of copper from 4 to 6 wt.% to Al yields strength near to that of sintered bronze. 95-5-Al-Cu powder mixture was compacted by Kemf(7) at 40 tsi (6.2 tonne/sq.cm.) and sintered for 4 hours at 550°C. He obtained a tensile strength of 32,330 psi (2264 Kg/cm^2) with an elongation of 2%. The attainable physical properties are shown in Table III. Cremer and Cardiano(7) have successfully sintered Al-4% Cu alloys at 600°C i.e. above the entectic temperature of these alloys, in spite of the usually observed tendency of alloy compact to slump or otherwise deform during such treatment. The beneficial effects of increasing the molding pressure on density and the increase in tensile properties with rising initial pressures are shown in Fig. II.4. The specimen were sintered at 600°C for 30 min. in H_2 , Air and N_2 as well and were quenched from 510°C immediately after sintering.

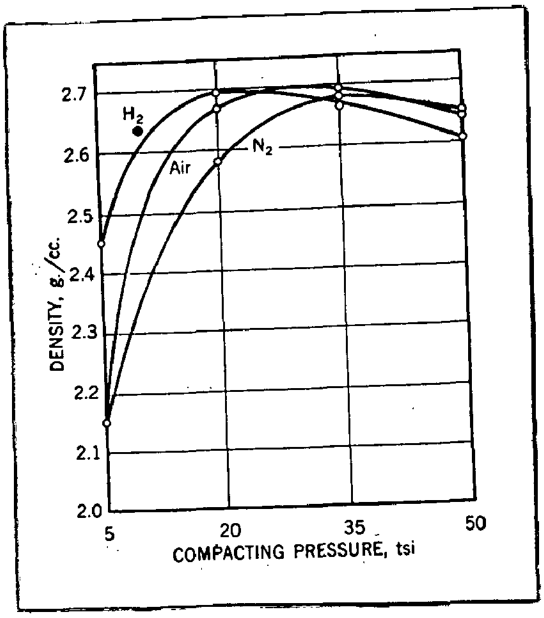


FIG. II.4 (a) EFFECT OF COMPACTING PRESSURE ON DENSITY OF COMPACTS PREPARED FROM 96-4 Al-Cu POWER MIXTURE AND SINTERED UNDER DIFFERENT ATMOSPHERES FOR 30 MINUTES AT 600°C (7)

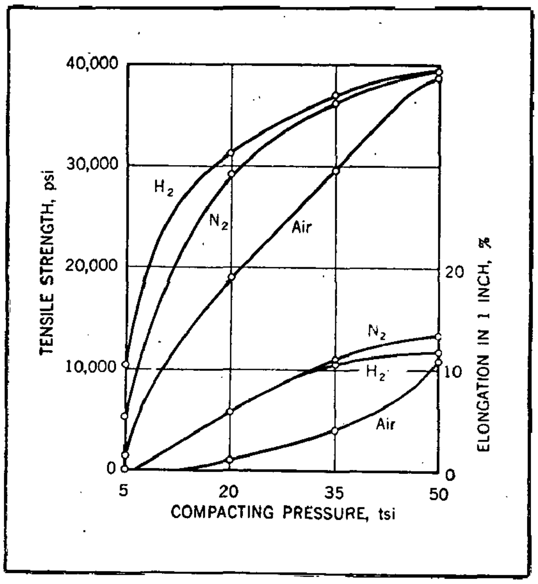


FIG.II.4(b) EFFECT OF COMPACTING PRESSURE ON TENSILE PROPERTIES OF COMPACTS PREPARED FROM A 96-4 Al-Cu POWDER MIXTURES AND SINTERED UNDER DIFFERENT ATMOSPHERE FOR 30 MINUTES AT 600°C (7)

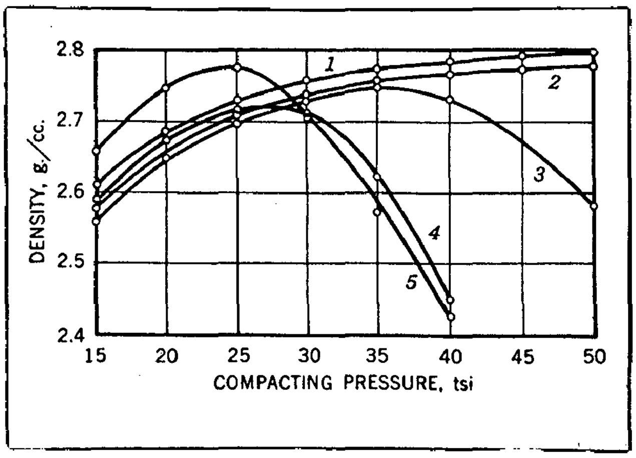


FIG.II.4(c) RELATION BETWEEN COMPACTING PRESSURE AND DENSITY AS AFFECTING BY SINTERING TEMPERATURE FOR 94-6 Al-Cu COMPACTS. ALL COMPACTS WERE SINTERED IN H_2 FOR 2 HOURS AT (1) 510°C (2) 528°C, (3) 560°C (4) 575°C (5) 590°C (8)

TABLE III : PHYSICAL PROPERTIES OF 95-5-Al-Cu COMPACTS

Sintering time hr.	Sintering atmosphere	Yield Strength psi	Tensile Strength psi	Elongation in 1 in. %	Reduction of area %
1	NH_3	15,300	19,700	4	1.6
	Air	15,600	19,100	3	1,8
4	NH_3	19,900	27,400	4	3.6
	Air	16,500	38,600	17	15.3

TABLE IV: PHYSICAL PROPERTIES OF ALUMINIUM -COPPER COMPACTS CONTAINING 6% Cu

Cold Pr. tsi	Sintering Temp. °C	Sintering Temp. °F	Furnace atmosphere	Brinell hardness	Ultimate strength psi	Elongation %
15	500	930	Air	43.6	12,000	<1.0
15	500	930	Vacuum	59.0	22,000	1.0
15	590	1090	Air	73.2	31,600	1.0
15	590	1090	Vacuum	88.7	40,200	5.0
50	500	930	Air	74.7	30,400	2.0
50	500	930	Vacuum	68.8	35,400	7.0
50	590	1090	Vacuum	91.8	48,000	7.0
5	620	1150	Vacuum	90.7	35,000	3.0
15	620	1150	Vacuum	110	36,200	3.0
50	620	1150	Vacuum	104	26,600	1.5

Bickerdike(8) sintered Al-6Cu in vacuum using colloidal graphite as a die lubricant. When sintering below the eutectic temperature the density was found to increase steadily with the compacting pressure. Compacts pressed at 20 tsi (3 tonne/sq.cm) from the mixture and sintered at 570°C for 2 hours exhibited a high strength of 42,600 psi (2955 kg/cm^2) with an elongation of 4.5%. A Al-6Cu compact cold pressed at 15 tsi (2.3 tonne/sq.cm) and then hot pressed at 5 tsi (.78 tonne/sq.cm.) and 500°C gave a BHN of 49.6, a tensile strength of 22000 psi (1540 kg/cm^2) and an elongation of 6%. The beneficial effect on Hardness and tensile properties sintered in vacuum as compared with sintering in Air become apparent from the data (Table IV).

The effect of compacting pressure and sintering temperature on tensile strength and elongation is shown in Table V, while the influence of copper particle size on hardness and tensile strength is shown in Table VI. All properties usually improve with rising sintering temperatures and with decreasing particle size of Copper component, whereas the trend with rising compacting pressure is not clearly marked, and for higher sintering temperatures optimum values are obtained for medium pressures.

Wantanabe and Yamada(9) studied Al-10% Cu alloys of 85% density. Using mixed, prealloyed and composite powders sintered at 560°C. They obtained maximum strength values with a +ve increase in volume in 1-2 hours and the mixed powders showed maximum effective strength of 17 kg/mm^2 . The sintering was carried out in the presence of the liquid phase, which appears because of the

ABLE V : EFFECT OF VARIATION IN COLD-COMPACTING PRESSURE AND SINTERING TEMPERATURE ON ULTIMATE TENSILE STRENGTH AND ELONGATION OF 94-6 Al-Cu COMPACTS

inter- ng emp.		Ultimate strength (in psi) for cold-compacting pressure in (tsi) of:							
°C	°F	15	20	25	30	35	40	45	50
10	950	-	-	16000	21000	27400	27800	31000	32800
28	982	-	-	19600	35000	38200	(28400)	39200	41000
60	1040	-	27800	42500	43600	44800	37000	-	23800
75	1067	23000	42600	39600	36000	1 3200	-	-	-
90	1094	42000	42600	40000	29400	-	-	-	-

inter- ng emp.		Elongation (in %) for cold-compacting pressure(in tsi) of:							
°C	°F	15	20	25	30	35	40	45	50
10	950	-	-	<1.0	1.0	2.0	3.0	3.5	5.0
28	982	-	-	<1.0	4.5	5.0	(1.5)	6.5	11.0
60	1040	-	<1.0	5.0	5.0	6.5	4.0	-	<1.0
75	1067	<1.0	4.5	7.0	4.0	1.0	-	-	-
90	1094	3.5	5.0	3.0	1.5	-	-	-	-

contact melting of the particles of dissimilar metals corresponding to the eutectic systems. The maximum strength obtained in case of mixed powders is due to the fact that during formation of liquid phase in dissimilar partical boundaries the Al oxide film coating the powder particle is broken.

The microhardness ofmetals and alloys in Al-10 Cu compact using mixed powders during sintering is shown in Table VII and Fig.II.5 gives the liquid phase percent in mixed prealloyed and composite powders during sintering.

Martsunova et al(10) worked on fine Al powders of 5 to 15 μ size alloyed with < 50 μ Cu, pressed at 10-50 KN/cm^2 (1.2 - 5.8 tonne/sq.cm.) and sintered in temperature range 460-640°C in silica tubes at a pressure of 1.33 N/m^2 (.014 tonne/sq.cm.) for 1 hou r. 1-5 wt.% of Cu in Al was investigated. The densification was found to increase with increase in pressure only till 30 KN/cm^2 (3.48 tonne/sq.cm.) and remained constant beyond this. 95% of the green density of the theqretical was obtained at about 30 KN/cm^2 (3.48 tonne/sq.cm.). Cu percentage has an influence on the porosity of green compacts because of its different formability and elastic recovery properties as compared to Al. The sintered density too depends upon the Cu content. At higher pressure the the contraction is obtained only in alloys with lower Cu content, and this increases with the increase in sintering temperatures. Initial and final porosites relations with compacting pressure Cu composition and sintering temperatures are shown in Figs.II.6, 7 and 8.

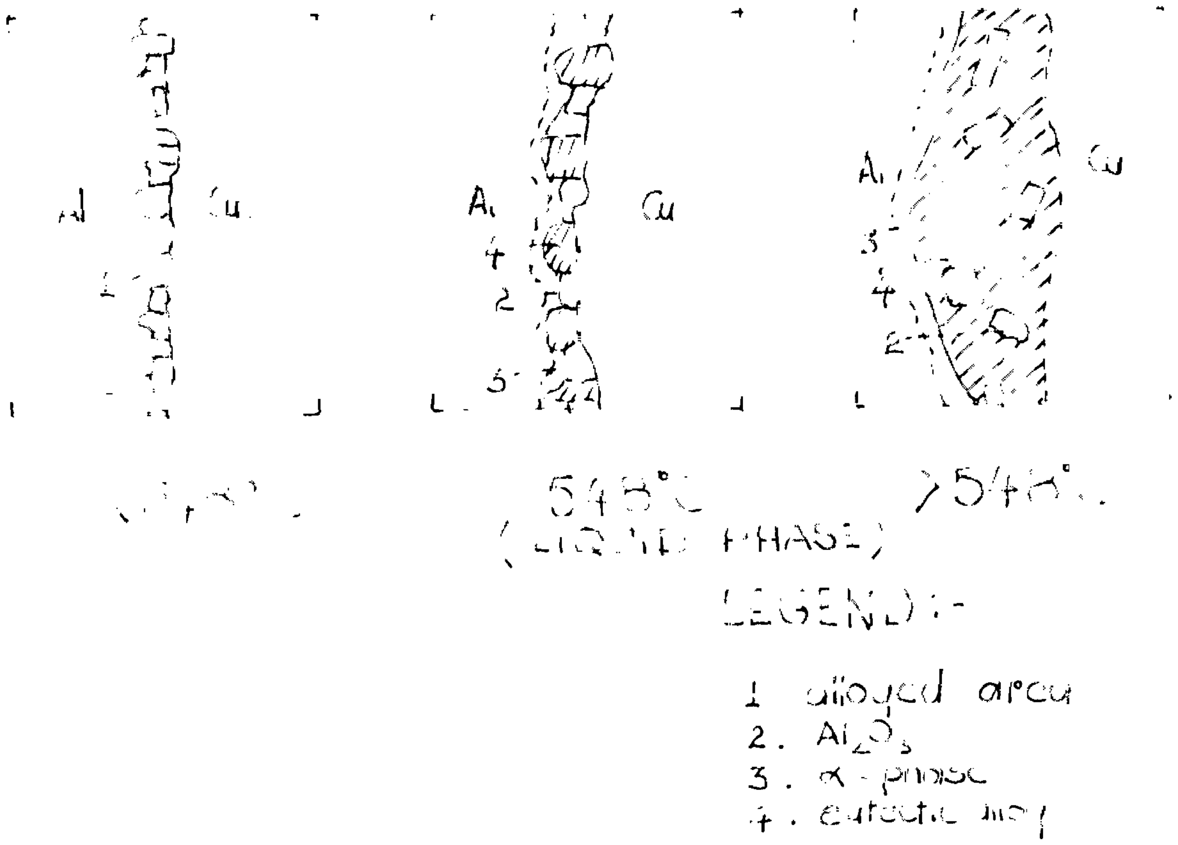


Fig. II-5
APPEARANCE OF LIQUID PHASE ON THE BOUNDARY OF ALUMINIUM AND COPPER POWDERS IN THE COMPACT, IN THE SURROUNDING OF THE EUTECTIC TEMPERATURE 548°C.
[illegible]

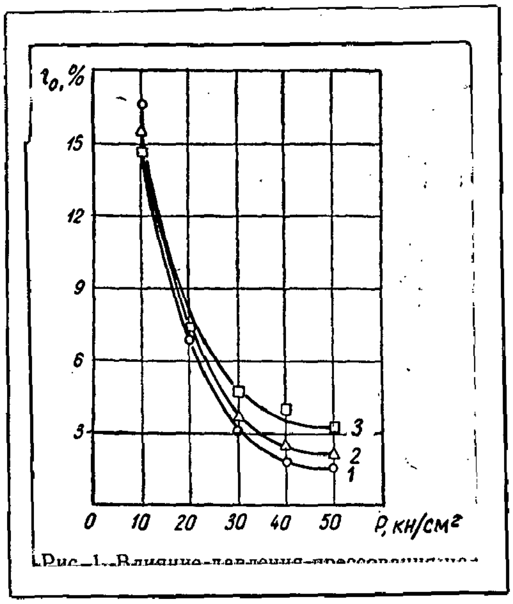


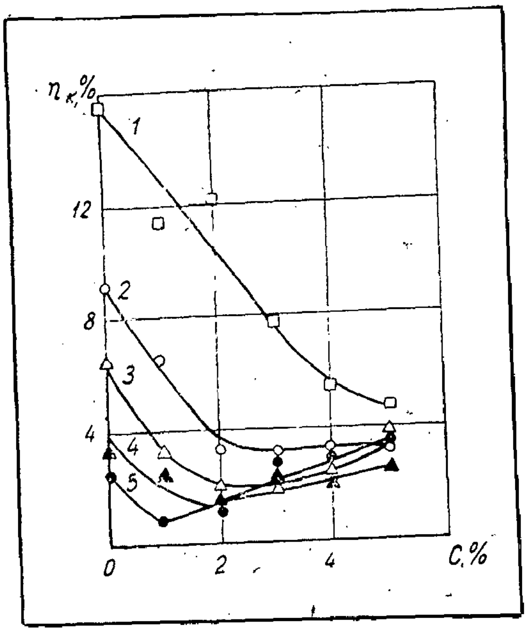
FIG.II.6

EFFECT OF COMPACTING PRESSURE ON THE POROSITY OF GREEN COMPACTS FOR DIFFERENT Al-Cu POWDER MIXTURES

i. WITHOUT ANY ADDITION
ii. 2 WT.% Cu
iii. 5 WT.% Cu (10)

FIG.II.7

DEPENDENCE OF POROSITY OF SINTERED COMPACTS, W.R.T. Cu COMPOSITION; SINTERING TEMPERATURE 640°C COMPACTING PRESSURE; 1. 1.2, 2. 2.32; 3. 3.46; 4. 4.64; 5. 5.58 TONNES/SQ.CM. (10)



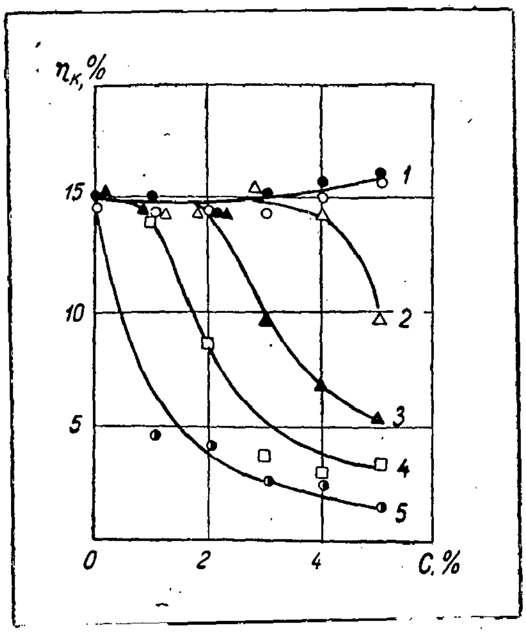


FIG.II.8

EFFECT OF Cu ADDITION ON FINAL POROSITY OF SINTERED ALLOYS COMPACTING PRESSURES; 1.2 TONNES/SQ.CM.; SINTERING TEMPERATURE
1. 460°C; 2. 580°C; 3. 600°C
4. 620°C; 5. 640°C (10)

TABLE VI : INFLUENCE OF COPPER PARTICLE SIZE ON HARDNESS AND ULTIMATE TENSILE STRENGTH OF SINTERED 94-6 Al-Cu COMPACTS

Copper particle size µ	Sintering Temp.		Brinell hardness	Ultimate tensile strength psi
	°C	°F		
< 45	520	968	71.7	26,200
45 - 79	520	968	64.3	19,600
79 - 160	520	968	51.5	14,800
< 45	552	1026	76.7	39,400
45 - 79	552	1026	59.0	36,200
79 - 160	552	1026	60.3	23,000

TABLE VII : CHANGE IN MICROHARDNESS OF METALS & ALLOYS IN Al-10% Cu COMPACT USING MIXED POWDER DURING SINTERING

Hardness	Metal & Alloys	Green Compact	Heated upto 560°C	Heated at 560°C for 300 min.
Micro-Vickers Hardness HV (50g)	Al	34 ± 1	29 ± 1	almost disappeared
	Cu-Al Alloy (α)	-	95 ± 3	91 ± 1
	Copper	60 ± 4	disappeared	-
	eutectic alloy(α + θ)	-	12 ± 3	116 ± 6

The author suggests a compacting pressure of 30 KN/cm^2 (3.0 tonne/sq.cm.). The hardness increases with increase in Cu content and/or sintering temperature. Very high temperatures may of courseyield coarse grains, which is because of the crystallization from the melt whose quantity in the alloy per volume is higher than the solid phase e.g. from 45% (3% Cu) to 75% (5% Cu) close to 640°C. The composition of phase precipitated or separated from the melt corresponding to the solid solution of Cu in Al is determined by the sintering temperature.

Maximum hardness of the alloy is obtained by such an alloying of Al with Cu at which the structure remains fine. However, the hardness is sufficiently high even when the structure of alloy becomes coarse. The hardness plot has been shown in Fig.II.9. X-ray analysis revealed that the lattice constant is decreased during dissolution of Cu in Al. Higher the sintering temperature till the appearance of liquid phase more and more Cu is dissolved in Al by means of solid state diffusion. The lattice constants of alloys in the temperature range of liquid phase sintering must increase with increase of sintering temperature. On observations of all the sintering temperature intervals they concluded that the curves corresponding to lattice constants of alloy w.r.t. sintering temperature have minimum.

The method of sintering above entectic temperature can be studied by seeing the Al-Cu phase diagram Fig.II.10. When heated above 548°C, the Al and Cu particles completely disappear and there appears a large amount of eutectic alloy ($\alpha + \theta$) having a

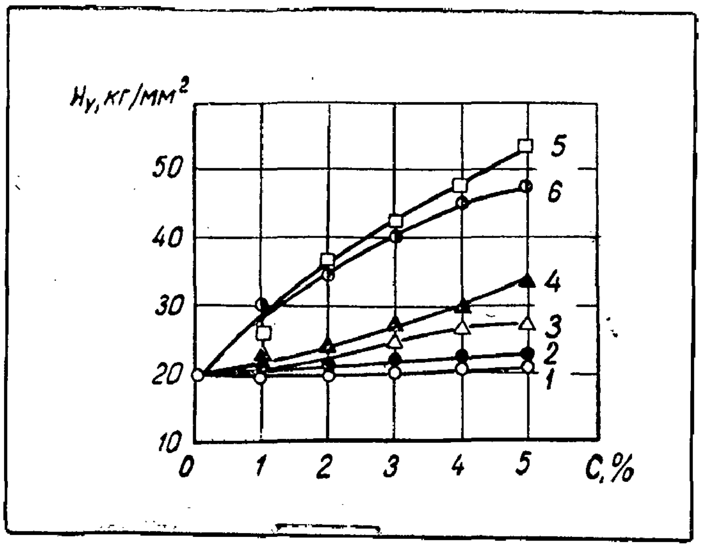
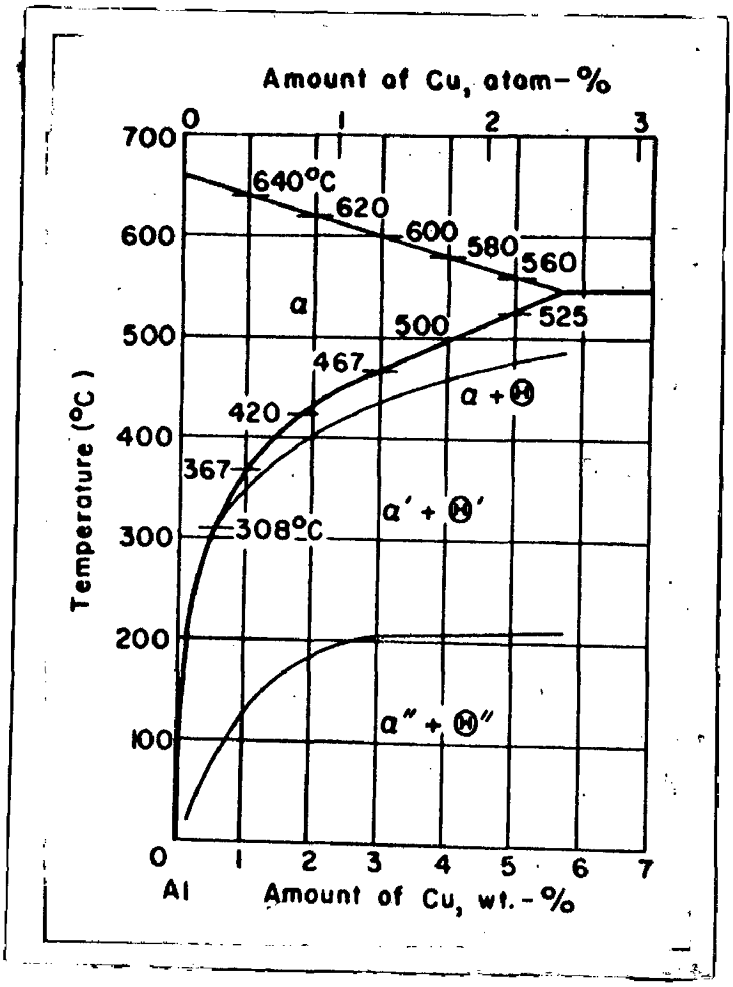


FIG.II.9

EFFECT OF Cu COMPOSITION ON HARDNESS OF Al-Cu ALLOYS SINTERED AT 1. 460°C; 2. 500°C; 3. 560°C; 4. 580°C; 5. 620°C 6. 640°C (10)

FIG.II.10

ALUMINIUM COPPER BINARY EQUILIBRIUM DIAGRAM SHOWING METASTABLE PHASES (25)



melting point of 548°C. The appearance of this liquid at just above the eutectic temperature and approaching a maximum value (33%) until the beginning of a steady 560°C stage, after which it should gradually decrease along with time and temperature to near the minimum value 10%. X-ray studies reveal that the part around the eutectic alloy become Al-Cu alloy (α)by diffusion of Cu atoms from the eutectic alloy, but the greater part of the remainder maintains a state, close to pure Al. At higher sintering periods the structure approaches a state of equilibrium by further diffusion of Cu atoms, that is, the eutectic alloy decreases, while nearly all the Al matrix become Al-Cu (α)alloy.

The alloying is restricted by the presence of Al_2O_3 scale. Thus it depends upon the metallic content of both powders following breakage of Al_2O_3 scale of the Al powder in the compacting press. On raising the temperature a small amount of eutectic alloy becomes liquid. Subsequently the liquid phase becomes the nucleus around which a large amount of liquid is rapidly produced. This is believed to be caused by the great rapidity in the diffusion of Al-Cu atoms into the liquid phase compared to the solid. This large quantity of liquid produced on the point of contact helps to scatter the Al_2O_3 scale and thus imparts high strength values. It is therefore desirable that the liquid appears in the neighbourhood of the Al_2O_3 scale and moreover it should be in large quantities. In the green compacts using mixed powders the Al and Cu powders already have metallic contacts through the cracks of Al_2O_3 scale. When this compact is heated, Al and Cu powder start alloying at comparatively low temperatures along this area of metallic contact, and as a result small amount of eutectic alloy is formed. Above the eutectic temperature the alloy becomes liquid, which by forming nucleii increases rapidly. This phenomenon continues till the Cu powder disappears. Because of this large quantities of liquid phase, the Al_2O_3 scale along the particle boundary scatter into liquid phase resulting in marked increase in strength of the compact. When kept at 560°C for a long time the copper atoms diffusing into the Al matrix cause the low strength eutectic alloy to decrease in amount and almost all the Al-matrix to become Al-Cu alloy (oc) possessing high strength so that the strength value depends upon the amount of liquid phase appearing in the compact, when the eutectic temperature is exceeded, and the position of its appearance. Mixed powders develop more liquid phase over pre-alloyed and composite ones and hence develop maximum strength.

F.J. Esper and G. Leuze(11) investigated Al-4.4 Cu compressed to 95% density, sintered in a muffle furnace with N_2 as protective atmosphere at dew point of -45°C to -65°C at 590°C for 30 min. Dilatometery was used to study the sintering process. It was found that the size of porosities decreases with decreasing particle size of alloying elements. The variation of Aluminium particle size did not give any significant results. The pore size is also effected in the same way. The dialation was caused above 540°C. Since 548°C is the eutectic temperature in Al-Cu Binary system. This temperature at which the dialation begun depended upon the particle size of Cu powder. The finer the copper powder, the lower is the temperature at the beginning of dialation. The amount of dialation is determined by the particle size of Al and Cu powders, and the influence of Al powder is stronger than that of Cu powder. It is seen that the liquid phase causes the dialation. The particles of a fine powder are very quickly liquified, so that the big dialation begins at a temperature compared with a coarse Cu powder. Since a small particle size of Cu and Al enables the liquid to disappear rapidly by diffusion the total dialation of the same is lowest. Therefore the amount of dialation decreases with increasing fineness of Cu and Al particles The tensile strength also increases significantly with decreasing particle size of Cu and varied from 2.0 k/mm^2 (200 kp/cm^2) to 10.5 kp/mm^2 (1050 kp/cm^2) as the powder particle size of Cu varied from -250 um to -32 µm respectively. It was correlated that as the pure size of structure decreased tensile strength increased. The dialatometer curves for Al-Cu alloys are shown in Fig. II.11 and 12.

II.3.2 Duralumin Type Alloys of Cu:

Compositions containing 10 and 20% Cu established(12) that in alloys in a state of partial equilibirum obtained by controlled sintering process, the properties are much dependant not only upon the degree of diffusion but also upon the presence of impurities. The ternary additions in Al-Cu systems have hence been investigated. Cremer and Cardiano(8) included 95-4-0.5-0.5 Al-Cu-Mg-Mn composition. On sintering and further heat treating they obtained hardness value of 50 RB (Fig.II.13). The specimens here pressed at 50 tsi (7.8 tonnes/sq.cm.) and sintered in dry N_2 atmosphere for 30 minutes and at 580°C and gave a tensile strength of 33000 psi (2310 Kg/sq.cm.) and elongation in 1 in of 10%. These compacts were water quenched at 510°C after sintering at 580°C.

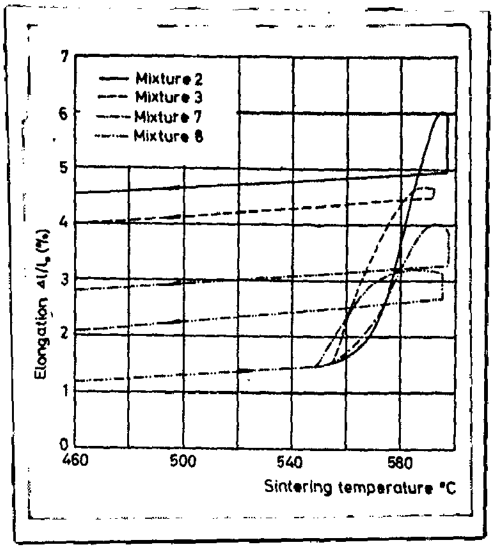


FIG.II.11

DILATOMETER CURVES FOR ALUMINIUM-COPPER MIXTURES. (11)

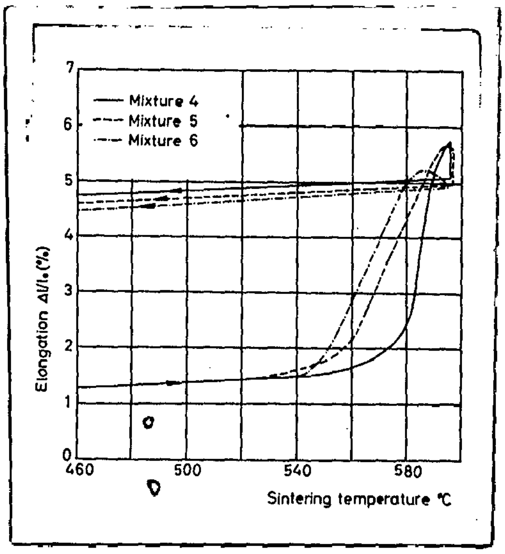


FIG.II.12

DILATOMETER CURVES FOR ALUMINIUM-COPPER MIXTURES. (11)

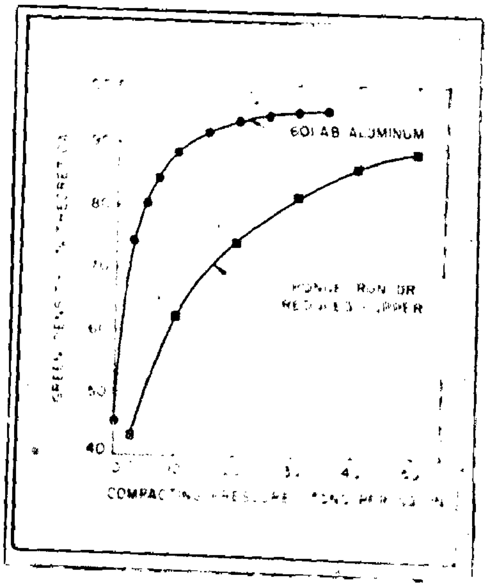


FIG.II.13

COMPRESSIBILITY CURVES OF ALUMINIUM-COPPER 601 AB ALLOY IN COMPARISON WITH COMPRESSIBILITY OF REDUCED COPPER (13)

For 97-2.5-0.5 Al-Cu-Mg compositions produced by compacting the elemental powder mixture at only 30 tsi (4-7 tonnes/sq.cm.) followed by sintering at 610°C for 30 minutes in N_2 atmosphere they report a tensile strength of 40000 psi (2800 kg/sq.cm.) with an elongation of 20% in the sintered state.

Systems containing Silicon in Al-Cu have also been investigated and render well to sintering and precipitation hardening.

Aluminium Corporation of America (AlCoa) has investigated systems containing .25% Cu, 4% Cu, 4.4 % Cu alloys, added to generate low melting intermetallic phases. The other additions are Mg, Si, Zn and other combinations, their compacting and sintering processes depend upon the composition, particle size and shape. The compressibility curve(13) for Al is compared to sponge iron or reduced Cu in Fig.II.13. Al-Cu parts have a high degree of reproductivity hence for the control accurate sintering temperature is suggested(14). It is observed that at higher densities amount of shrinkage decreases. Density is affected more at higher temperatures of sintering. The effects of density and temperature on dimensions is shown in Fig.II.14. Dew points and atmospheres too effect the dimension changes. Higher dew point yield in expansions of compacts. Thus there would be a sharp decrease in properties. The dimensional cracks provide a +ve indication of furnace dew point conditions. The effect of dew point on dimensions and tensile strength are shown in Fig.II.15. The atmospheric effects of N_2 , dissociated NH_3 and vacuum have been reported(15) Best sintering properties are obtained in N_2 . The results are shown in Figs. II.16-18.

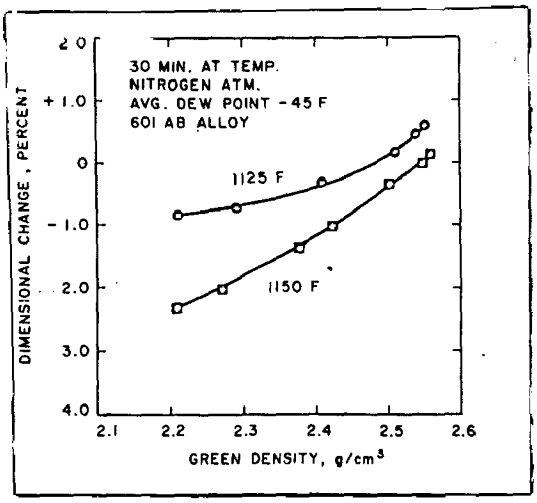


FIG.ii.14

EFFECT OF DENSITY AND TEMPERATURE ON DIMENSIONS. (13)

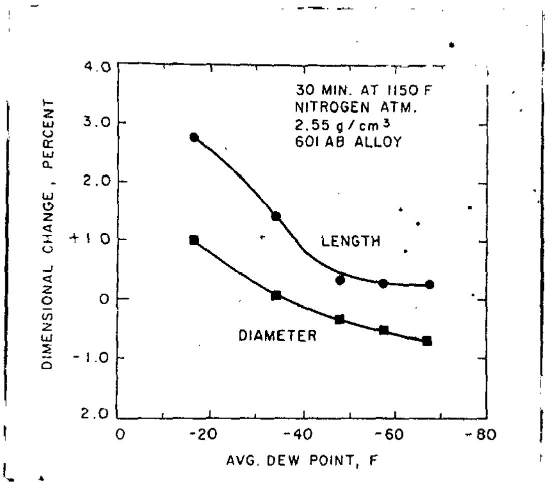


FIG.II.15(a)

EFFECT OF DEW POINT ON DIMENSIONS. (13)

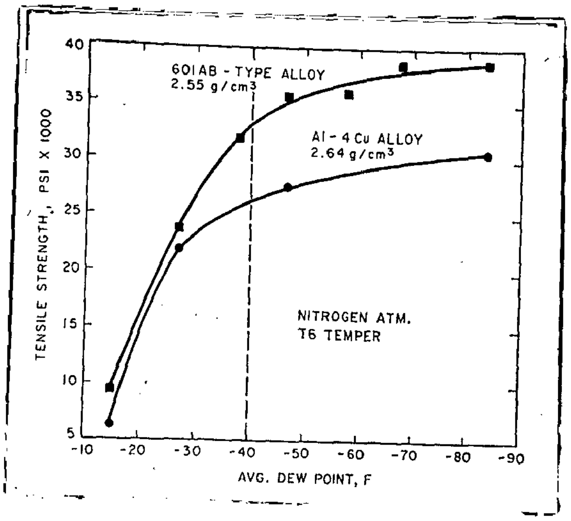


FIG.II.15 (b)

EFFECT OF DEW POINT ON TENSILE STRENGTH (13)

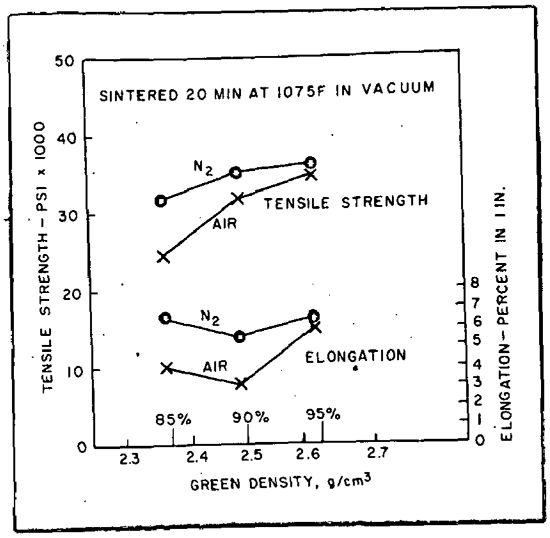
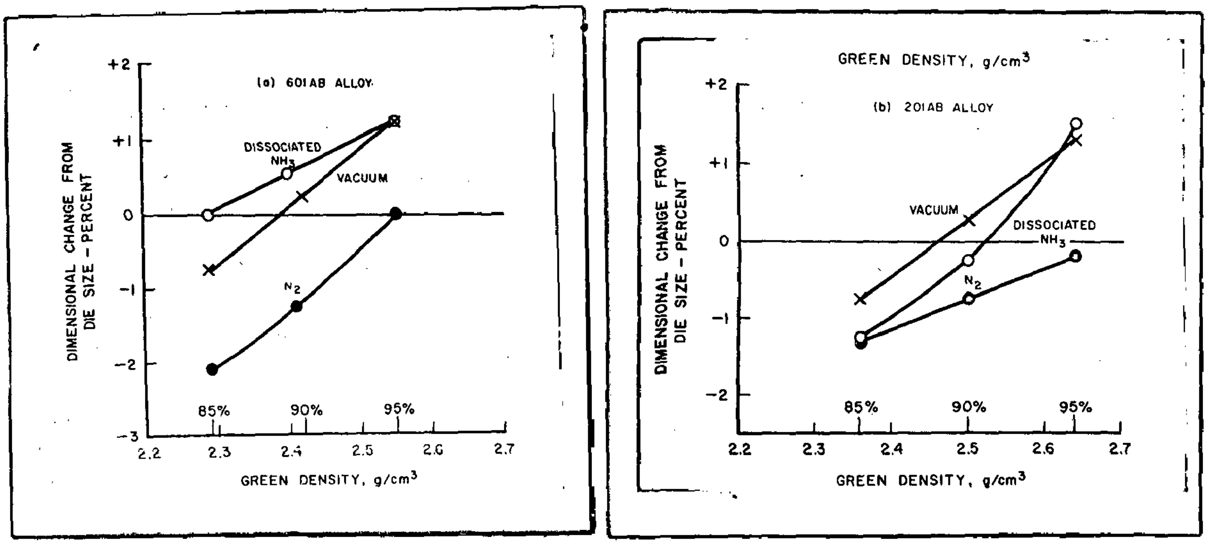


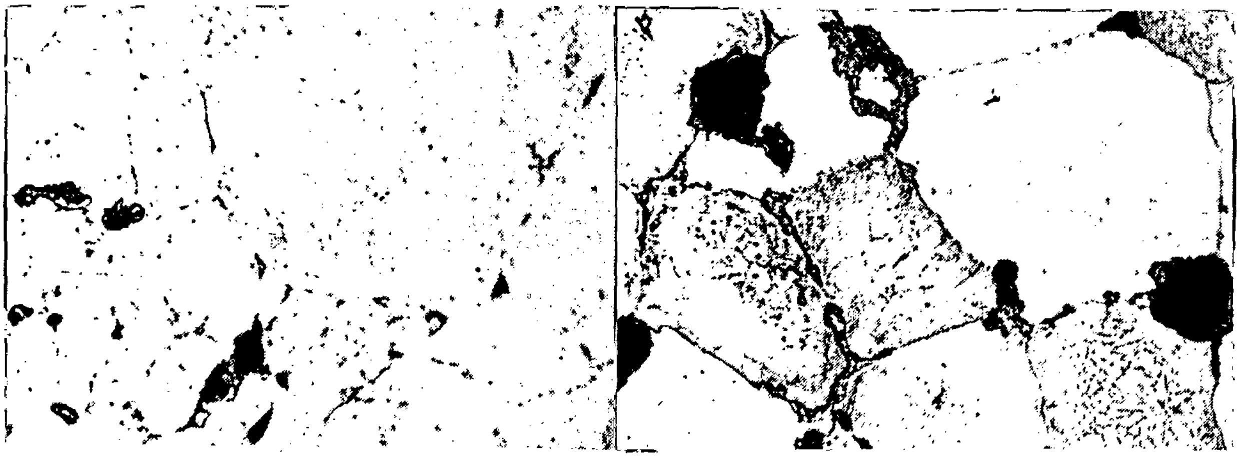
FIG. II.16

EFFECT OF PRESINTERING ATMOSPHERE ON PROPERTIES OF 201 AB-T_4 SINTERED IN VACUUM. (15)



(a) (b)

FIG.II.17 EFFECT OF GREEN DENSITY AND ATMOSPHERE ON SINTERED DIMENSION OF (a) 601 AB AND (b) 201 AB ALLOYS (15)



(a) (b)

FIG.II.18 PHOTOMICROGRAPHS SHOWING MICROSTRUCTURES OF SINTERED (a) 601 AB AND (b) 201 AB ALLOYS MAG. 500 X, (REDUCTION 50%) (15)

0.25% of Cu alloys are designated by Alcoa as 601 AB 601 AC and 602 AB commercial alloys. Their compositions are given in Table VII (a). The blends with Ac suffix contain no lubricant and are used for isostatic pressing. The Al powder 1202 is the base material in these alloys. The particles are irregular and nodular in shape which permit good particle interlocking during compacing to provide high green and sintered strength. The alloying additions have also been added to increase the sintered strength. These have been compacted at low pressures and obtain good densit ies at comparatively low pressures. At 12 tsi 601 AB has been compacted to 90% theoretical density and to 95% density at 25 tsi (3.9 tonne/sq.cm.). The compression ratio ranges from 1.5 to 1.9 :: 1 depending upon compacting pressure and the method of feeding. The sintered properties have been detailed in Table IX and green density of 601 AB in Table X (a). 601 AB alloy achieves a tensile strength of ranging from 16000 to 36000 psi (1120 to 2520 kg/sq.cm) depending upon the density and heat treatment. Hot forging at 427°C followed by heat treatment gave(16) tensile strength of 32000 psi to 38000 psi (2240-2660 kg/cm^2) ultimate and 20,000 psi (1400 kg/cm^2) yield along with 6-16% elongation (in 1 in.) with 601 AB-T_4 and 44,000 to 50,000 psi (3080 - 3500 kg/cm^2) ultimate, 44,000-46,000 psi (3080 to 3220 kg/cm^2) yield and 0.8% elongation with 601 AB-T_6 depending upon forging pressure and %age reduction during forging. Hardness has been used as an approximate indication of strength for Al p/m parts. The value ranges from 55-60 Rockwell H for them. The hardness test was considered particularly beneficial as a control check during processing.

TABLE VIII : NOMINAL COMPOSITION OF Alcoa ALLOYS

(a) .25% Cu Alloys

Blend	Cu	Si	Mg	Fe	Al	Lubricant
601 A B	.25	.6	1.0	.3 max.	1202 powder	1.5
601 AC	.25	.6	1.0	.3 max	1202 powder	-
602 AB						

(b) Duralumin Type Alloys

Blend	Cu	Si	Mg	Fe	Al	Lubricant
201 AB	4.4	.8	0.4	.3 max.	1202 powder	1.5
201 AC	4.4	.8	0.4	.3 max.	1202 powder	-
202 AB	4.0	-	-	-	1202 powder	1.5

TABLE IX : DESCRIPTION OF VARIOUS TEMPERS FOR SINTERED Al P/M ALLOYS

(a)

Alloy	Green Density %	Green Density g/cm^3	Thermal Condition	Tensile Strength MN/m^2	Yield Strength MN/m^2	Elongation % in 2.5 cm
601 AB	90	2.42	T_1	121	55	7.0
			T_4	-	-	-
			T_6	224	214	2.0
Sintered 30 min at 621°C in N_2						
	95	2.55	T_1	124	58.4	8.0
			T_4	152	103	5.0
			T_6	252	241	2.0
601 AC	90	2.42	T_1	137	54.5	11.0
			T_4	187	102	9.3
			T_6	267	260	1.3
Sintered 20 min. at 593°C						
	95	2.55	T_1	134	53.1	10.7
			T_4	189	98.5	10.0
			T_6	269	261	2.0
602 AB	90	2.42	T_1	121	58.4	9.0
			T_4	121	62	7.0
			T_6	179	169	2.0
Sintered 15 min. at 635°C						
	90	2.42	T_2	138	121	3.5
			T_4]	134	76	9.0
			T_6]	183	169	2.5
	95	2.55	T_1	131	62	9.0
			T_4	134	65.5	10.0
			T_6	186	172	3.0
	95	2.55	T_2	141	114	5.0
			T_4]	141	79.5	10.0
			T_6]	193	176	3.0

TABLE IX : DESCRIPTION OF VARIOUS TEMPERS FOR SINTERED Al P/M ALLOYS (b)

Alloy	Green Density %	Green Density g/cm^3	Thermal condition	Tensile strength MN/m^2	Yield Strength MN/m^2	Elongatio % in 2.5 cm
201 AB	90	2.50	T_1	210	170	3.0
			T_4	246	206	3.5
			T_6	323	-	-
Sintered 30 min. at 593°C in N_2						
	95	2.64	T_1	228	181	3.0
			T_4	262	214	3.5
			T_6	332	327	2.0
201 AC	90	2.50	T_1	183	136	3.3
			T_4	286	223	4.7
			T_6	337	-	
Sintered 20 min. at 593°C in N_2						
	95	2.64	T_1	205	147	4.0
			T_4	319	230	6.0
			T_6	382	370	2.0

TABLE X (a) : GREEN DENSITY AND STRENGTH OF 601 AB BLENDED ALLOY

Compacting Pressure	Green Density g/cc	Green Density %age	Green Strength psi
7	2.29	85.0	450
12	2.42	90.0	950
25	2.55	95.0	1500

TABLE X (b):

Compacting pressure	Green Density g/cc	Green Density % age	Green Strength psi
7	2.33	83.7	550
12	2.49	89.5	1150
25	2.63	94.5	1900

Repressing improved the results, on repressing at 25 tsi (3.9 tonne/sq.cm.) the density was improved from 2.45 g/cc to 2.58 g/cc, along with a 4000 to 5000 psi (280-350 kg/cm^2) increase in tensile strength.

Alcoa 202 AB grades contain 4% Cu(17). Itis easily mechanically or isostatically compacted and is specially suitable for application in parts requiring high strength coupled with high ductility. The relation of compacting pressure with green density of Al-Cu blend is shown in Fig.II.19 and their properties in Table XI.

Alcoa 201 AB and 201 AC are 4.4% Cu ternary alloys having nominal composition as in Table VIII (b). Their green densities is shown in Table X (b). 201 AB develops higher strength levels upto 48,000 psi (3360 kg/cm^2) in the fully heat treated T_6 temper. Properties of 52,000-58,000 psi (3640-4060 kg/cm^2) ultimate, 37,000-38,000 psi (2590-2660 kg/cm^2) yields and 8-18% elongation have been achieved with 201 AB-T_4 and 57,000-63,000 psi (3990-4410 kg/cm^2) ultimate and 56,000-60,000 psi (3920-4200 kg/cm^2) yield and .5 to .8% elongation with 201 AB-T_6. After cold forging the tensile properties have been evaluated as 26,000-50,000 psi (1820-3500 kg/cm^2) ultimate strength, 13,000-48,000 psi (910-3360 kg/cm^2) yield strength and 1-13% elongation.

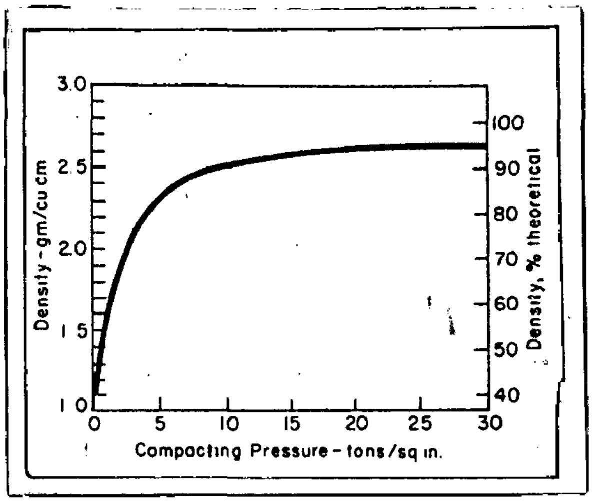


FIG. II.19

COMPACTING PRESSURE VS GREEN DENSITY OF ALUMINIUM- 4% COPPER BLEND (1.5% LUBRICANT) (17)

TABLE XI : GREEN AND SINTERED PROPERTIES OF 202 AB COMPACTED AND COLD FORMED PARTS

Green Density %	Green Density gm/cc	Green Str. psi	Sintered Density %	Sintered Density gm/cc	Dimen. change %	Heat treated	Ten. Str. 1000 psi	Yld Str. 1000 psi	Comp. Yld str. 1000 psi	Elong %	App. Hardness R_E
Compacted Performs:											
90	2.5	510	95	264	-2.6	T_1	23.2	10.9	-	10.0	-
						T_4	28.2	17.2	-	8.0	-
						T_6	33.0	21.3	-	7.3	-
Cold formed parts:											
90	2.5	510	95	2.64	-2.6	T_2	33.9	31.4	33.4	2.3	80
						T_4	34.3	21.5	-	8.0	70
						T_6	39.8	25.1	20.6	8.7	85
						T_8	40.6	36.2	-	3.0	87

Note: Presinter 3 hr. at 700-800°F in N_2. Sinter 15-30 min. at 1165 in N_2 with avg. dew point -45°F.

T_1 As sintered cooled to 800°F (426.7°C) in N_2 air cooled to 6'°F

T_4 Solution heat treated 30 min. at 1000°F (537.8°C) cold water quenched aged 4 days at 68°F = 19°C.

T_6 Same as T_4 except aged 20 hr. at 300°F (148.9°C)

T_8 Same as T_4 except immediately cold formed after water quench and then aged 20 hr. at 300°F (198.9°C)

Test results obtained with sintered Al specimens are compared with the physical properties of cast and wrought pure Al in Table XII.

TABLE XII : PHYSICAL PROPERTIES OF CAST, WROUGHT AND SINTERED Al

Material	Pressure tsi	Sintering cycles	Density % of Theoretical	Tensile Strength psi	Elong. %	Red. area %
Cast Al	-	-	-	8000 - 12000	20 - 40	30 - 60
Wrought Al	-	-	-	10000 - 15000	35 - 45	50 - 70
Sintered Al from -30 mesh powder	10	615°C 1 hr cracked NH_3	88.0	9900	33	37
	20		95.4	11810	47	59
	30		98.0	11850	48	57
	40		98.8	11790	48	59
	50		99.5	11900	46	55
Sintered Al from -100 mesh powder	10	-do-	85.0	9700	37	44
	20		94.2	11810	47	63
	30		97.3	12000	49	66
	40		98.4	11970	48	63
	50		99.0	11900	46	60

It is particularly interesting to note the high ductility values found in sintered metal. Indicating that not only refractory metals, but also low melting basically ductile metals can be produced by P/M to high degree of ductility provided that they are practically free of pores.

II.4 Activated Sintering of Al and its alloys:

No work on the activated sintering of Al-Cu alloys is available. The activation effect may be well achieved by chemical method of activation e.g. in the presence of very small quantities of alloying elements. The possible groups in this case are:

i. Use of alloying elements with mutual solubility.

ii. Use of alloying elements that do not have solubility in the substrate. The substrate however has large solubility in the added element.

An activating effect of character i) depends largely on the chemical nature, mobility, and mode of introducing the alloying element. The second group system is characterized by three important aspects:

a. The added metal is almost insoluble in the base metal.

b. The base metal must to some extent be soluble in added metal, so that it must diffuse through it.

c. The added metal should not oxidise through sintering.

The operating mechanism is as when a solid solution exists the diffusion process is inhibited due to higher energy formation of vacancies. In the studies of solid solution formed during sintering, a strong sinterability was observed with increasing thickness of second element layer. By analogy with the enhanced chemical reacticity of phases in the nascent state, an increased capacity for sintering is readily conceivable in solid solution. This phenomenon is interpretted in terms of difference in partial diffusion coefficients that exists in almost all binary systems and results in an accumulation of vacancies on one side. The efficacy of the activating additives depends not only on the nature of added elements, but also on the method of its introduction. During sintering alloying of contact area between the particles of compact is accomplished by other metals which may be an additional source of the properties of sintered material.

It has been found that use of very small quantities of alloying elements lowers the sintering temperatures considerably. Theoretical densities can be achieved in a relatively shorter time. Thus a sintering process is termed to be activated(19) if

i. The activation energy required to transform a particulate system into a coherent body is lowered, i.e. the diffusion path is changed favourably.

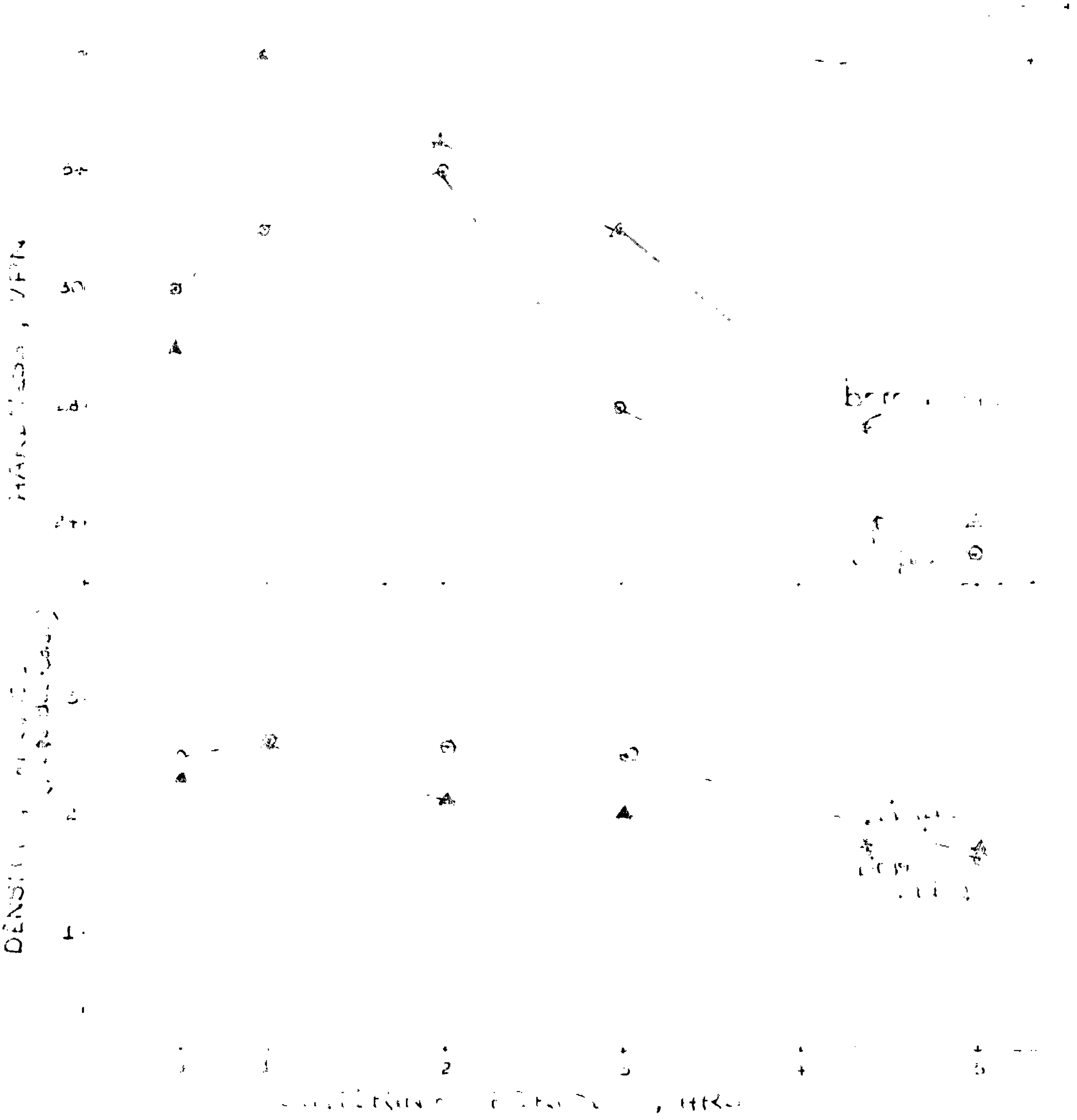
ii, The number of atoms participating during mass transport is increased i.e. diffusion flow is increased.

iii, The surface or interparticle contact are improved.

The small percentages of added alloying elements may influence as follows: the non-uniformities caused by additives effect the sintering(20) behaviour of an array of particles for creating a diffusion gradient much larger than that normally considered in the derivation of sintering equations. Since perfect atomic mixing is generally not achieved upon the mechanical mixing of powders, the impurities or additives appear at the surfaces of host powders, and therefore at the necks of sintering particles. If there is a larger tendency for the host material to diffuse into the impurity than the additive out, of the impurity then the concentration gradient from the mass centre of the particle to the neck may be rate controlling. At elevated temperatures changes in solid solubility will change the driving force too. When the compacts are first heated, surface impurities are still highly concentrated at the necks and a faster shrinkage may be expected. As the shrinkage proceeds, much alloying occurs and the driving force is greatly diminished. This would be the case if the impurities or additive, remained in the neck. If the impurity diffused rapidly out from the neck, sintering to high densities would be highly impaired. This would cause the transport of matter away from the neck. When the impurity content is high Kirkendall type of diffusion could result in the pores in the neck. Both of these would inhibit neck formation, growth and densification. Any possible improvement in the host material's bulk diffusivity by the formation of defects would be concentrated by a residual neck porosity, and a subsequent decrease in grain boundary diffusivity. This inhibits pore annihilation. Thus it is expected that even a small concentration of additive can not always be neglected, and may even play a dominent role in the sintering process.

Author(21) studied the activation effect on Al-3% Cu alloys by boron following the method of chemical addition. Copper was chosen as second element in order to study its diffusion character in the Al-Cu systems. It was aimed as to improve the sintered propeities under same conditions, or reducing sintering period and/or temperature with practically no changes in properties. -45 µm of Al, Cu and B powders were compacted at 5.1 tons/sq.cm. to give a green density of 85% of simple Al-Cu and 80% for Boron added alloy compacts. These were sintered at 550°C in vacuum of the order .05 mm of Hg. The results are shown in Fig.20.

Decrease in density i.e. swelling was observed on sintering.



[illegible] II. [illegible]
[illegible]
[illegible]
[illegible] 5 [illegible]
[illegible] ; 550°C. (21)

The effect of Boron was two fold:

1. It shifted the peak hardness value to left i.e. increased the rate of homogenization by increased diffusion between Al and Cu particles.

2. Overall hardness was more in case of boron added specimens. The density was found to be constant till 4 hours of sintering and a change was noted at the fifth hour. This suggests that the porosity content started decreasing only after 4 Hours of sintering (This could be true as oxide film of Al delays the bond formation between Al particles). Grain growth and hence softening in structure was indicated after one hour in Boron added and after two hours in simple Al-Cu sintered compacts. It was concluded that the Boron activated the process.

II.5 Scope of the Present Work:

Although aluminium can be formed into low cost, close tolerance parts by other processes such as extrusion and die casting yet there are a number of cases where powder Metallurgy is the ideal process. In Table XII different test results obtained with sintered aluminium specimens are compared with the physical properties of cast and wrought pure aluminium. It is particularly interesting to note the high ductility values found in sintered metals. This indicates that if the part ~~it~~ can be produced practically free of pores by P/M, it can achieve high degree of ductility and strength. Aluminium P/M parts are less costly than Al die casting e.g. because of lower die cost and shorter lead time. Due to its light weight, the transportation charges are also minimum. Thus the cost saving has averaged to 35.4% on substituting existing metals and manufacturing methods with Al sintered components(23). It has high malleability which provides ease in coming resulting in close size control and mirror like finish. Forgeability is also improved because sintered Al possesses a uniform structure without alloy segregation and grain orientation.

In the present investigation it was planned to develop Aluminium base copper alloy system by p/m, to study its sintering behaviour and the extent to which it can be activated. Previous work with 3% Cu-Al by author(21) had shown positive results of activation with Boron in Al alloy system, although the properties were not satisfactorily obtained. So the selection of sintering temperatures and other parameters have been re-established to optimise the sintering procedure.

CHAPTER III

EXPERIMENTAL PROCEDURE

III.1 Powder Characteristics:

III.1.1 Aluminium Powder:

Aluminium powder of 99.9% purity manufactured under arrangements with E Merck AG. Darmstadt (Germany) by Sarabhai M Chemicals Ltd., Baroda (Batch No. OC 00493) was used. Its characteristics are as under:

1. Sieve Analysis: BS S Sieves were used. Powders were shaken in sieve shaker for 45 minutes, and 78% powder was -53 μm.

2. Flow: The powder was heated at 110°C and stored in desicator. It did not flow through the Hall cup.

3. Apparent Density: It was measured by the ASTM. B 417-64. The powder was non-free flowing. The measured apparent density was 0.96 g/cc and a theoretical density value of 2.7 g/cc was used.

4. Particle Shape: The atomised aluminium particles revealed regular particle shape under the microscope, at a magnification of 600 X.

III.1.2 Copper Powder:

Electrolytic copper powder of 99.99% purity was used in the present investigation. The powder characteristics are as follows:

1. Sieve Analysis: Powder was shaken in BSS Sieves for 45 minutes. 99% of powder was -53 µm and 60% below 37 µM.

2. Flow: Dried powder did not flow through the Hall cup.

3. Apparent Density: The apparent density of this powder measured by ASTM B 417-64 method was 1.36 g/cc, the theoretical density value was taken as 8.92 g/cc.

4. Particle Shape: The particles were irregular in shape and dendritic under the microscope when seen at a magnification of 600 X.

III.1.3 Boron Powder:

Amorphous Boron powder was used in the present investigation It was ground in pestle mortar for 10 hours. Its particle size was reduced to 4 to 6 um. The ground powder had a spherical particle shape. A theoretical density value of 2.54 g/cc was taken in the theoretical density calculations of the alloy,

III.2 Blending:

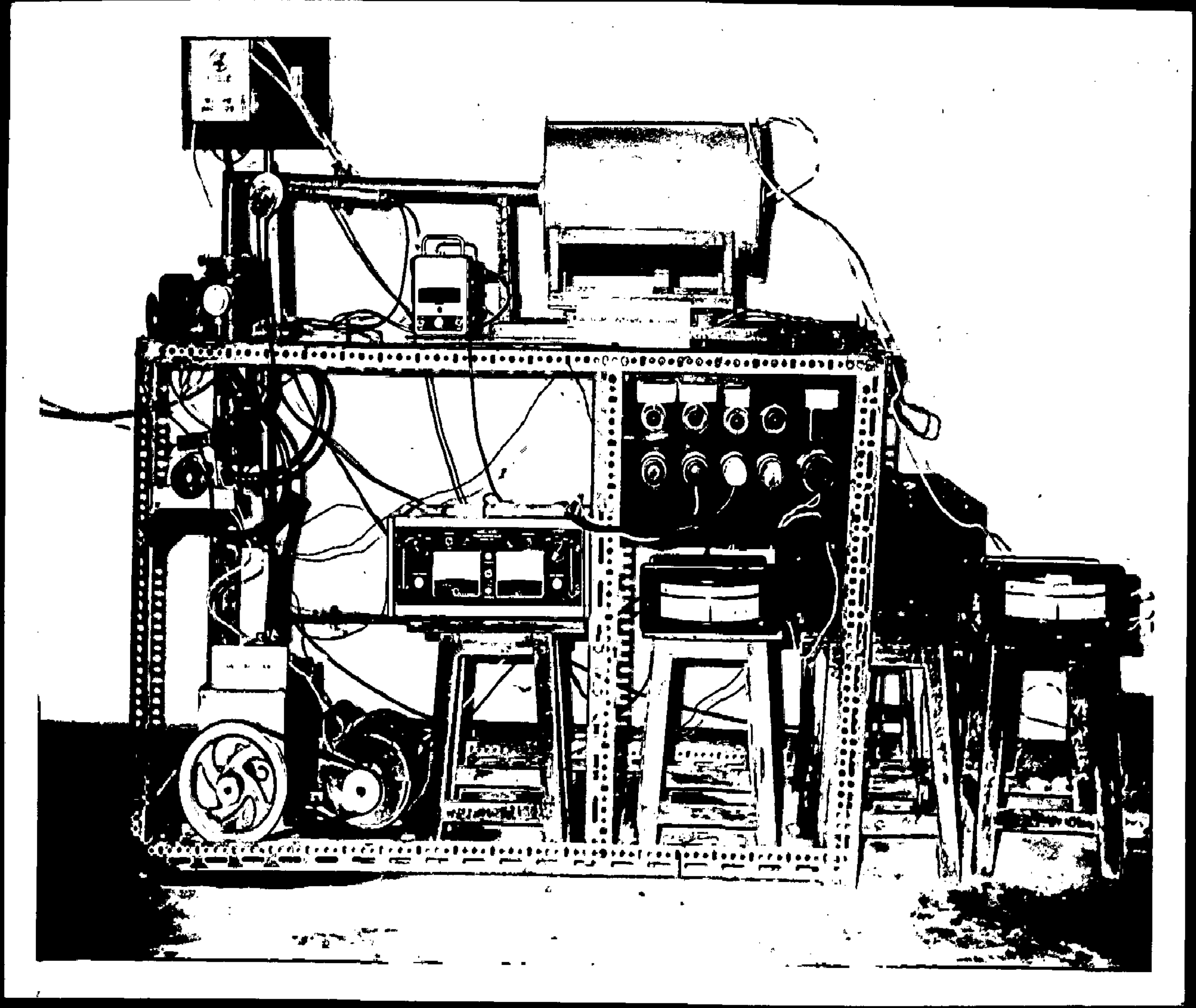
Three compositions were chosen viz. 3% Cu-Al, 4% Cu, 5% Cu. Four lots of Al, Al-Cu alloy, and another four of Boron added systems were made. These lots were blended in a double cone blender for 8 hours each till the uniform mixture was obtained. (The weighing was made to an accuracy of 4 decimal places). The powders were later stored in a desiccator.

III.3 Green Compact Formation:

High speed steel punch and die of internal dia 1.628 cm was used for the purpose. The die was cleaned with Zinc stearate to the welding of powder with the die walls. The cylindrical compacts were formed using 2.1 gm of respective powder in a hydraulic pressing machine with a load variation of ± 5 pc. The edges of green compact were smoothened by carefully rubbing on the fine emery paper to facilitate the correct dimensions measurements. The weights employed were 1.51, 2.26 and 3.02 tons/sq.cm. Desired compacting pressures were obtained in 1/2-1 minute and then kept constant for 1½ minute in each case. The release was made in 1/2 minute.

III.4 Sintering of Compacts:

The compacts were sintered in a vacuum of approximately 10^{-4} mm of mercury in a Kanthal wound resistance furnace. The sintering substrate in the compact was placed on stainless steel pedestal. The specimens were heated to a temperature in the range of liquids and solids on the equilibrium diagram i.e. at 600°C in one hour and then maintained for the required time. The sintering cycle is shown in fig . It was selected so as to drive off moisture, to volatile material etc. before the sintering temperature was achieved. The sintering time was measured from the instant the temperature was measured to within ± 5°C with a thermocouple placed just above the specimen. The sintering temperature was controlled very approximately to the set value, by setting the voltage to the furnace, at a constant value. After sintering the specimens were cooled in vacuum.



EXPERIMENTAL SET UP USED FOR VACUUM SINTERING OF ALUMINIUM ALLOY COMPACTS

A sintering temperature of 600°C was selected in order to obtain a liquid phase, as this temperature is above the entectic temperature i.e. 548°C on the equilibrium diagram of Al-Cu alloy. The percentage of liquid phase was < 1% in case of Al-3%Cu, 5.3 in case of Al-4% Cu and 10.5 in Al-5% Cu. Sintering periods of 5 mts 1 hour, 2 hours, 3 hours and 4 hours were chosen. Simple an d Boron added Al powder compacts were also sintered at 600°C which is essentially a solid phase sintering.

The sketch of the experimental unit employed for sintering is shown in Fig.III.1.

III.5 Density Measurement:

The density measurement was done by measuring the heights of the specimens at the opposite ends of a square, and along the diameter by a micrometer of least count .001 cm. and calculated. The theoretical densities data of Al as 2.7, Cu as 8.92 and Amorphous Boron as 2.54 were used in the work and the theoretical density of alloys was calculated with the formula

$$d_t = \frac{M_t\ d_x\ d_y}{m_x\ d_y + m_y\ d_x}$$

and

$$d_t = \frac{M_t \cdot d_x \cdot d_y \cdot d_z}{m_x \cdot d_y \cdot d_z + m_y \cdot d_x \cdot d_z + m_z \cdot d_x \cdot d_y}$$

where x,y and z are the different components. M_t the total mass of the powders. d_x, d_y, d_z, are the densities of the required components.

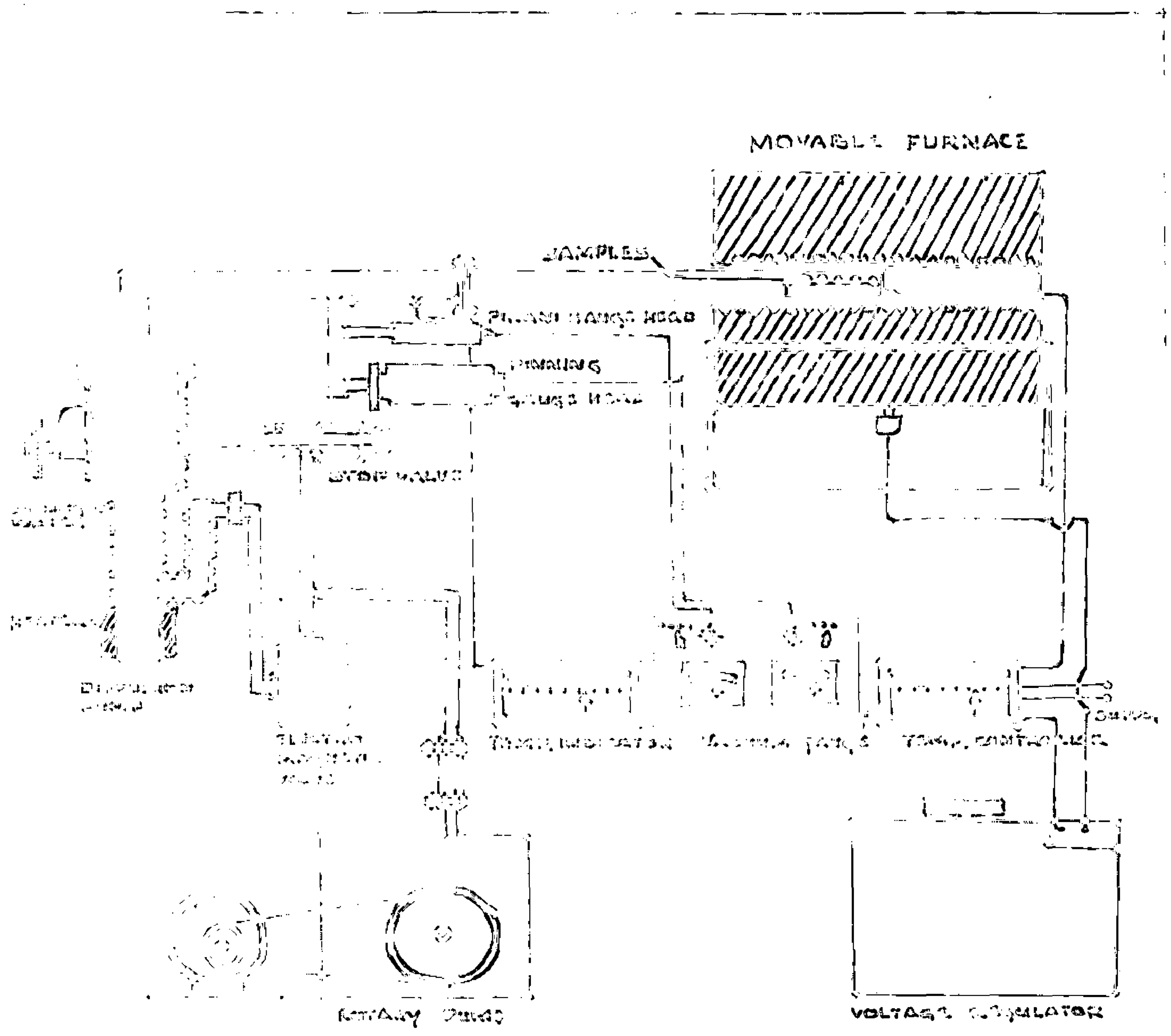


FIG. III.1 : SCHEMATIC DIAGRAM OF :- VACUUM SINTERING APPARATUS.

III.4.2. Densification Parameter:

The densification parameter (ΔD) was calculated by the formulae

$$\Delta D = \frac{D_S - D_O}{D_T - D_O}$$

where D_S = Sintered density

D_O = Green Density

and D_T = Theoretical density.

The densification parameter gives better understanding of changes in density while takes into account the real change in dimen sions and the green dimensions.

III.6 Hardness Measurement:

The hardness measurements were carried out on Vickers Hardness Tester at 5 Kg. load. The observations were taken along the diameter. These were in the range of ± 5 VPN. The medium of these readings was taken as hardness value of the concerned specimen

III.7 Microstructural Studies:

The specimens were polished per usual techniques. The microstructures revealed satisfactorily by etching with hydrofluoric acid (HF). Metallographic studies were conducted on the large incident light camera as "Neophot 2". The microstructures were examined at different magnifications through out specimen, but the microstructures were taken at a magnification of 150 X.

CHAPTER IV

RESULTS

IV.1 Effect of Compacting Pressure on Properties of Al-Cu Alloy

IV.1.1 Green Density:

Green density values of powder mixes investigated namely Al, Al-3% Cu, Al-4% Cu, Al-5% Cu compressed at pressures of 1.5, 2.3 and 3.0 tonnes/sq.cm. are shown in Fig.IV.1 (Table IV.1). The density of all compacts increases as compacting pressure increases, the change in uniform particularly in case of pure Aluminium. At a compacting pressure of 1.5 tonnes/sq.cm. the green density is decreased with an increase in the copper content but as the pressure is increased to 2.3 tonnes/sq.cm. the green density of Al-5% Cu alloy compact increased considerably and measures more than that of other two alloys.

IV.1.2 Densification Parameter:

Densification parameter values related to compacting pressures are shown in Fig.IV.2 (Table IV.2). The curves for aluminium rises with the pressure and then decreases giving a maximum at 2.3 tonnes/sq.cm. It is contrary in cases of alloys which give a minimum at the above pressure. The alloy mixers have almost the same densification parameter when compressed at the same pressure.

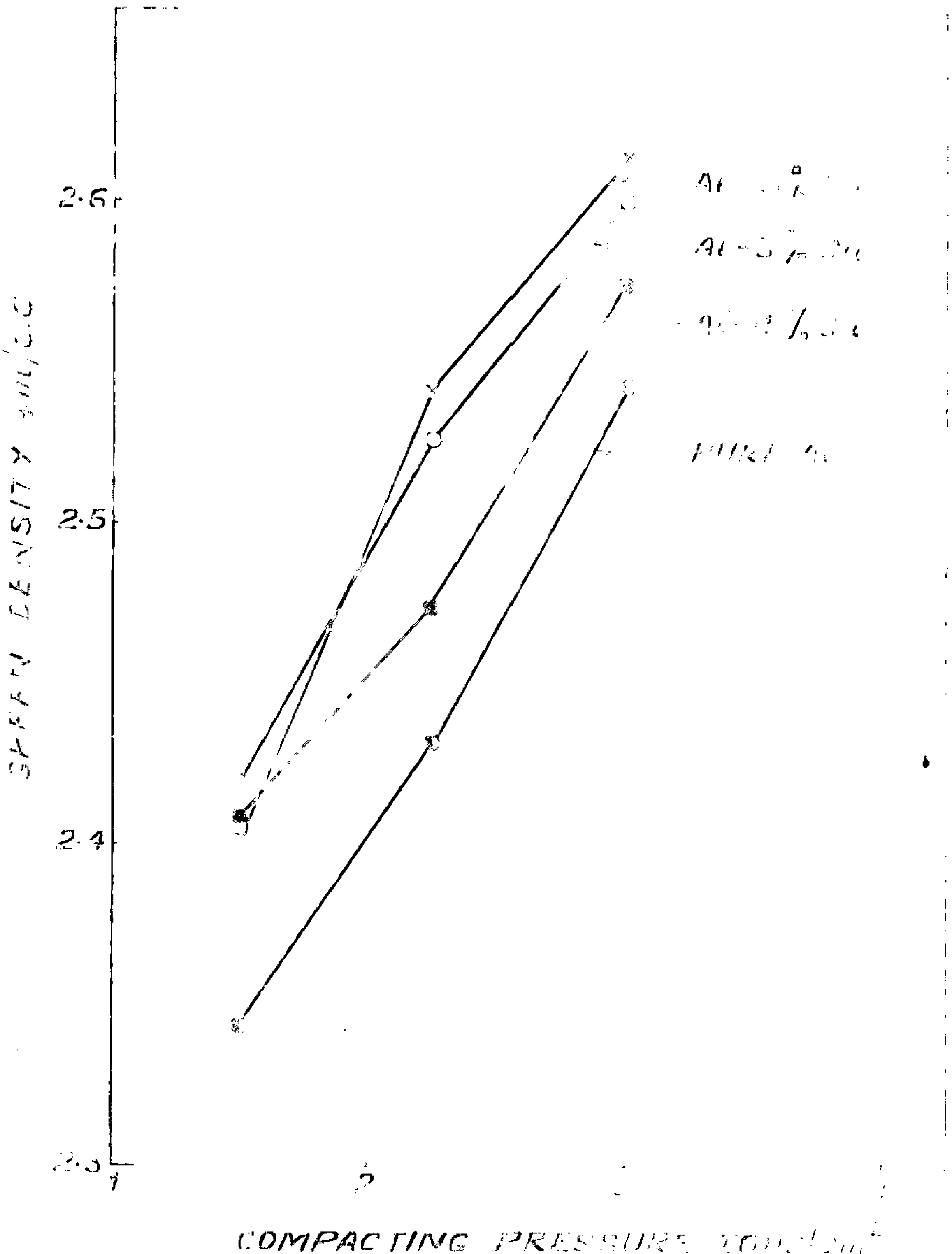


Fig. IV.1 Effect of Compacting Pressure on Green Density of (a) Pure Aluminium (b) Al-3% Cu (c) Al-4% Cu (d) Al-5% Cu

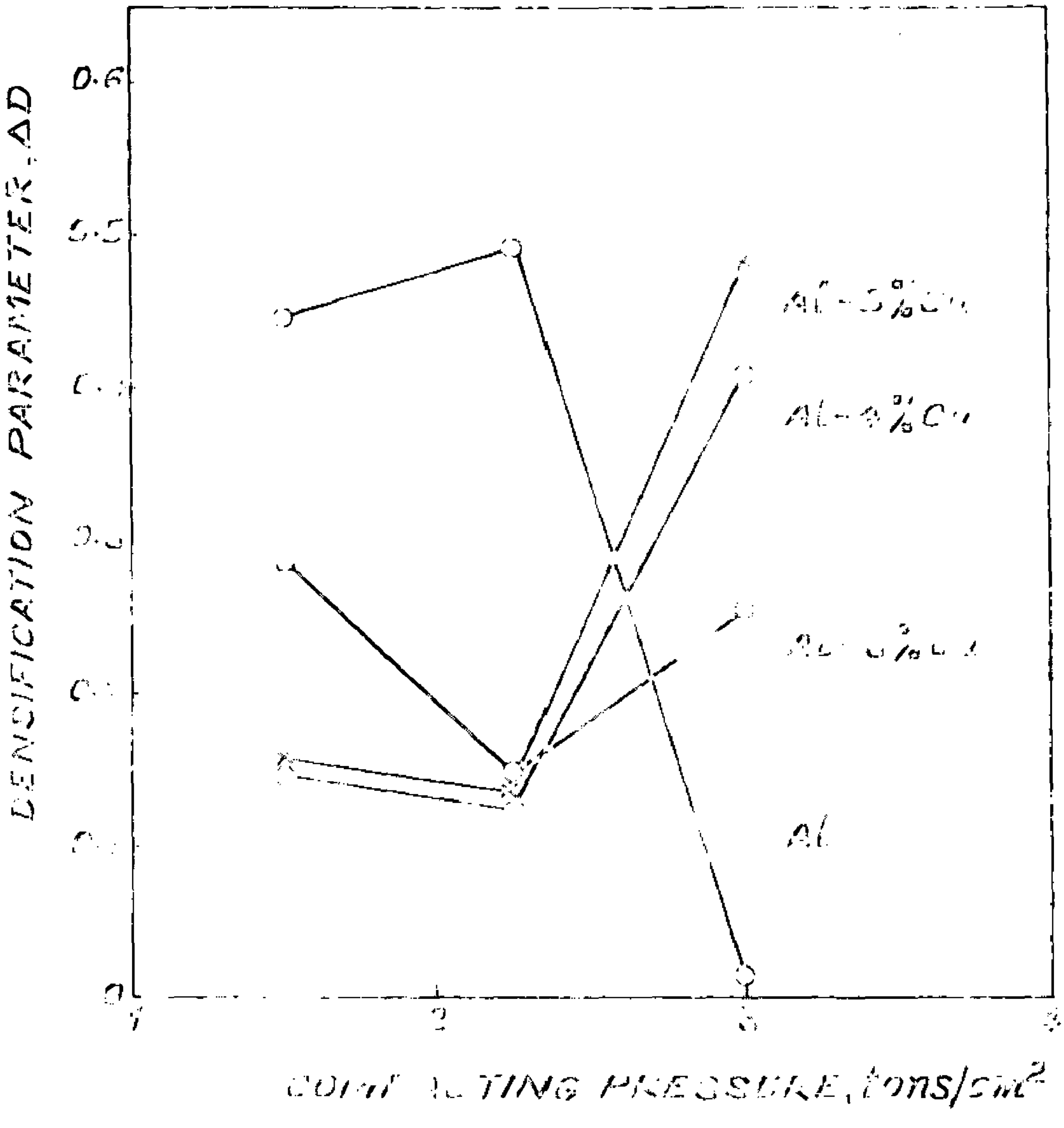


Fig. IV.2 Effect of compacting pressure on the densification parameter of (a) pure aluminium (b) Al-2% Cu (c) Al-4% Cu (d) Al-5% Cu alloys; sintered at 600°, for 1 hour.

TABLE IV.1: EFFECT OF COMPOSITION AND COMPACTING PRESSURE ON GREEN DENSITY OF Al-Cu ALLOYS

Composition	Green Density g/cc		
	1.51 tonnes/ sq.cm.	2.26 tonnes/ sq.cm.	3.02 tonnes/ sq.cm.
Al-powder	2.34	2.43	2.54
Al-3% Cu	2.42	2.53	2.60
Al-4% Cu	2.40	2.48	2.58
Al-5% Cu	2.40	2.54	2.61

TABLE IV.2 : EFFECT OF COMPOSITION AND COMPACTING PRESSURE ON DENSIFICATION PARAMETER OF Al-Cu ALLOYS, SINTERED AT 600°C FOR ONE HOUR

Composition	Densification Parameter ΔD		
	1.5 tonnes/ sq.cm.	2.3 tonnes/ sq.cm.	3 tonnes/ sq.cm.
Al - powder	0.449	0.049	0.0152
Al - 3% Cu	0.288	0.148	0.259
Al - 4% Cu	0.149	0.132	0.415
Al - 5% Cu	0.159	0.139	0.489

It is observed that practically no densification is obtained in Al when compressed at 3 tonnes/sq.cm. However, the curves reveal a very interesting fact i.e. when the compaction pressed at 3 tonnes/sq.cm. the densification parameter increases regularly in the order Al, Al-3%, 4%, 5% Cu alloys. The densification parameter (ΔD) values at 2.3 tonnes/sq.cm. compacting pressure suggest that in order to obtain constant dimensional changes, this compacting pressure of 2.3 tonnes/sq.cm. would be ideal for the further investigations.

IV.1.3 Hardness:

The variation of hardness with compacting pressure is shown in Fig.IV.3 (Table IV.3). Hardness increases with compacting pressure in cases the Cu content in Al is less than 5%. In Al-5% Cu alloy maximum hardness value is obtained when it is compacted at 2.3 tonnes/sq.cm. It is seen that as the Cu content in Al increases the hardness value of compacts increases but for Al-5% Cu system it is pronounced only upto compacting pressure of 2.3 tonnes/sq.cm.

IV.2 Effect of Boron Addition on Aluminium Alloy Systems:

The effect of Boron addition on Al and Al-Cu alloy systems for different sintering periods upto 4 hours is studied in the present investigation. The physical properties i.e. densification parameter and hardness are compared in Figs. IV.4-11, Tables IV.4, 5.

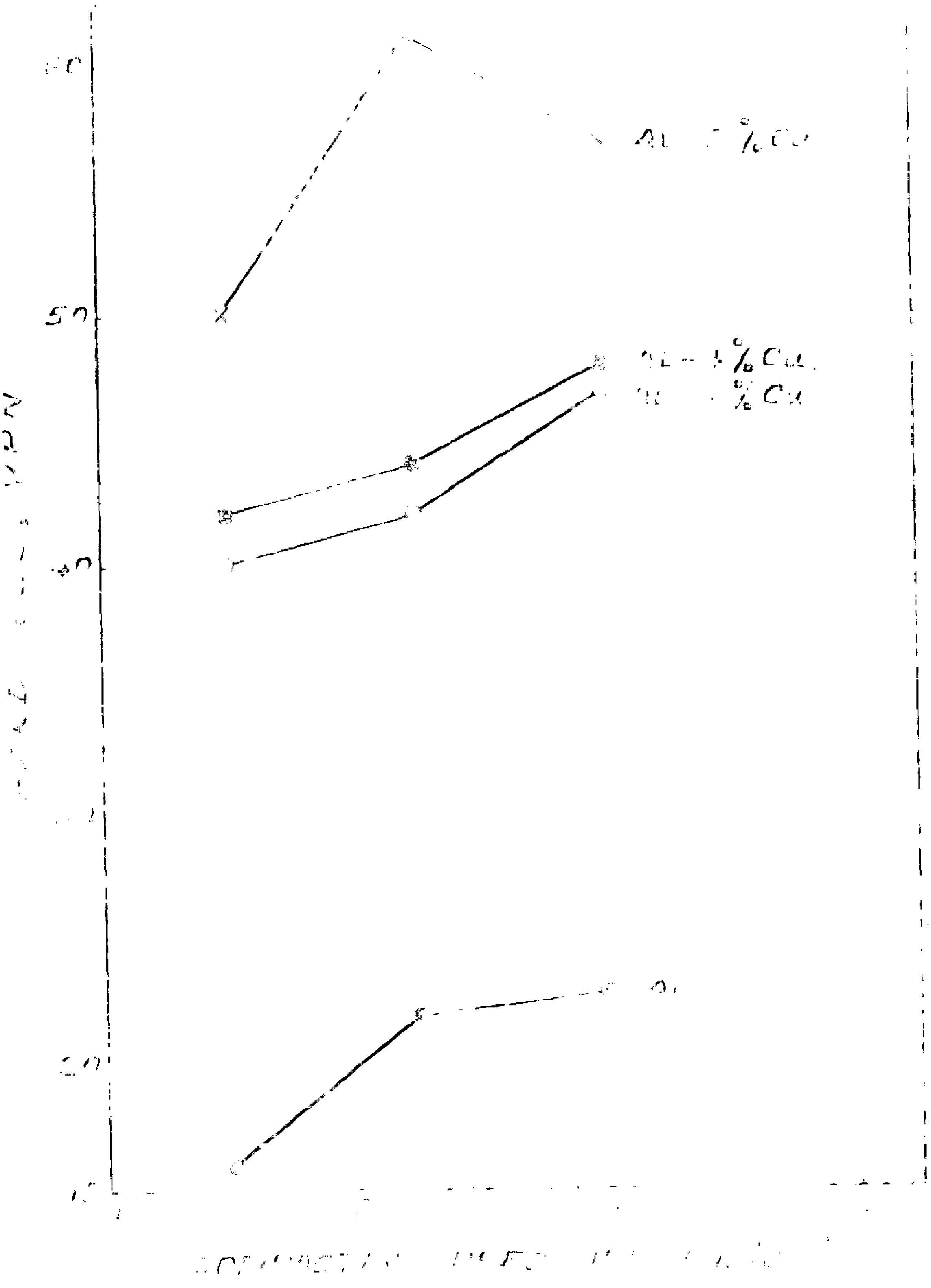


Fig. IV.5 Effect of Compacting Pressure on Hardness (a) Pure Aluminium (b) Al-3% Cu (c) Al-4% Cu (d) Al-5% Cu alloys; sintered at 500°C for 1 hour.

TABLE IV.3 : EFFECT OF COMPOSITION AND COMPACTING PRESSURE ON HARDNESS OF Al-Cu ALLOYS, SINTERING AT 600°C FOR ONE HOUR

Composition	Hardness (VPN)		
	1.5 tonnes/ sq.cm.	2.3 tonnes/ sq.cm.	3 tonnes/ sq.cm.
Al - Powder	16	23	24
Al - 3% Cu	40	42	47
Al - 4% Cu	42	44	48
Al - 5% Cu	50	61	57

TABLE IV. 4 : EFFECT OF COMPOSITION AND SINTERING PERIOD ON DENSIFICATION PARAMETER OF Al-Cu ALLOYS SINTERED AT 600°C

(a) SIMPLE

Alloy	5 mts.	1 hr.	2 hrs.	3 hrs.	4 hrs.
Al	0.018	0.047	0.025	0.021	0.018
Al - 3 wt% Cu	0.131	0.148	0.157	0.309	0.366
Al- 4 wt% Cu	-0.016	0.132	0.125	0.272	0.226
Al - 5 wt% Cu	-0.280	0.139	0.112	0.252	0.158

(b) BORON ADDED

Alloy	5 mts.	1 hr.	2 hrs.	3 hrs.	4 hrs.
Al	-0.007	0.051	0.052	0.000	-0.022
Al - 3 wt% Cu	-0.007	0.193	0.225	0.251	0.261
Al - 4 wt% Cu	-0.214	0.262	0.280	0.286	0.140
Al - 5 wt% Cu	0.292	0.337	0.373	0.350	0.234

IV.2.1 Densification Parameter:

In the case of Aluminium (Fig.IV.4) Δ D is positive in Boron activated compacts till 2.30 hours and negative later, as compared to simple Al powder. However, expansion of Boron added Al compacts is observed when sintered for five minutes. Again expansion in Boron added specimen was marked at a higher sintering period of four hours. The Δ D value at three hours was zero for Boron added compact. Maximum densification in case of simple Aluminium was obtained when sintered for one hour and it is decreased as sintering period is increased. As the Cu content in Boron added Al increases, longer sintering periods could be made possible to obtain a positive densification as compared to simple Al-Cu compacts. For Boron added Al-3% Cu, as compared to simple Al-3% Cu, a positive Δ D value is obtained till two hours sintering period, which (positive Δ D value) increases to three hours for activated Al-4% Cu and to four hours(throughout positive) for Boron added Al-5% Cu compositions. Expansion in case of Boron added alloy mixtures containing 3% and 4% copper was observed when sintered for a period of 5 minutes. But contraction was observed in case of Al-5% Cu-.05% B compacts sintered for a period of 5 minutes. For Al-3% Cu the contraction remained constant till 2 hours and then increased, but this increase was small and gradual after a sintering period of 1 hour. For simple Al-4% Cu compacts the contraction increases till 3 hours and then decreases whereas in case of Boron added samples there is a constant contraction when sintered between 1-3 hours, ΔD values decreases later. In Al-5% Cu compacts expansion is observed in

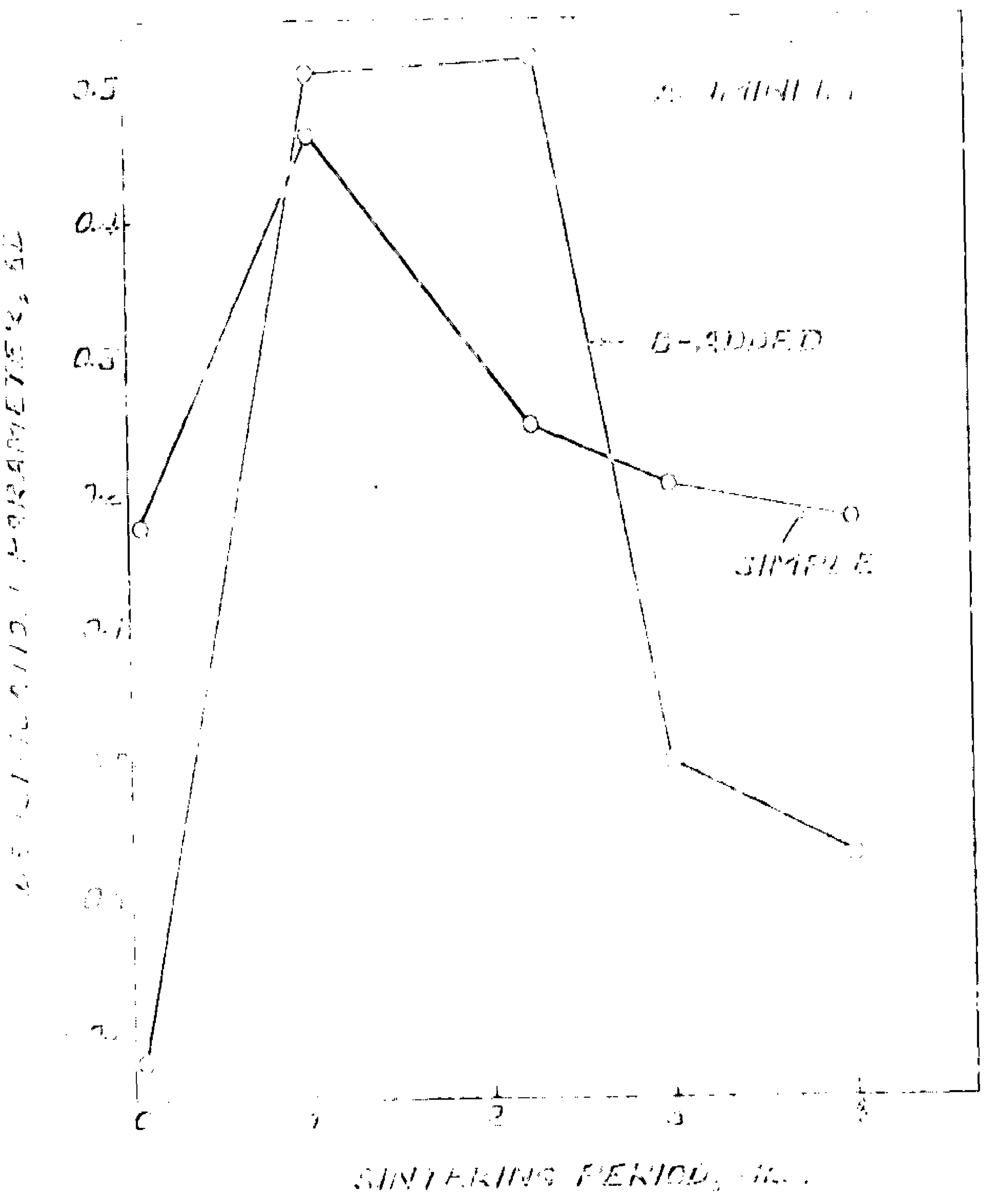


Fig. IV.4 Effect of sintering period on the [illegible] strain parameter of [illegible] powder compacts (a) simple, (b) boron added (0.05 wt.%); compacting pressure 2.5 tonnes/cm^2

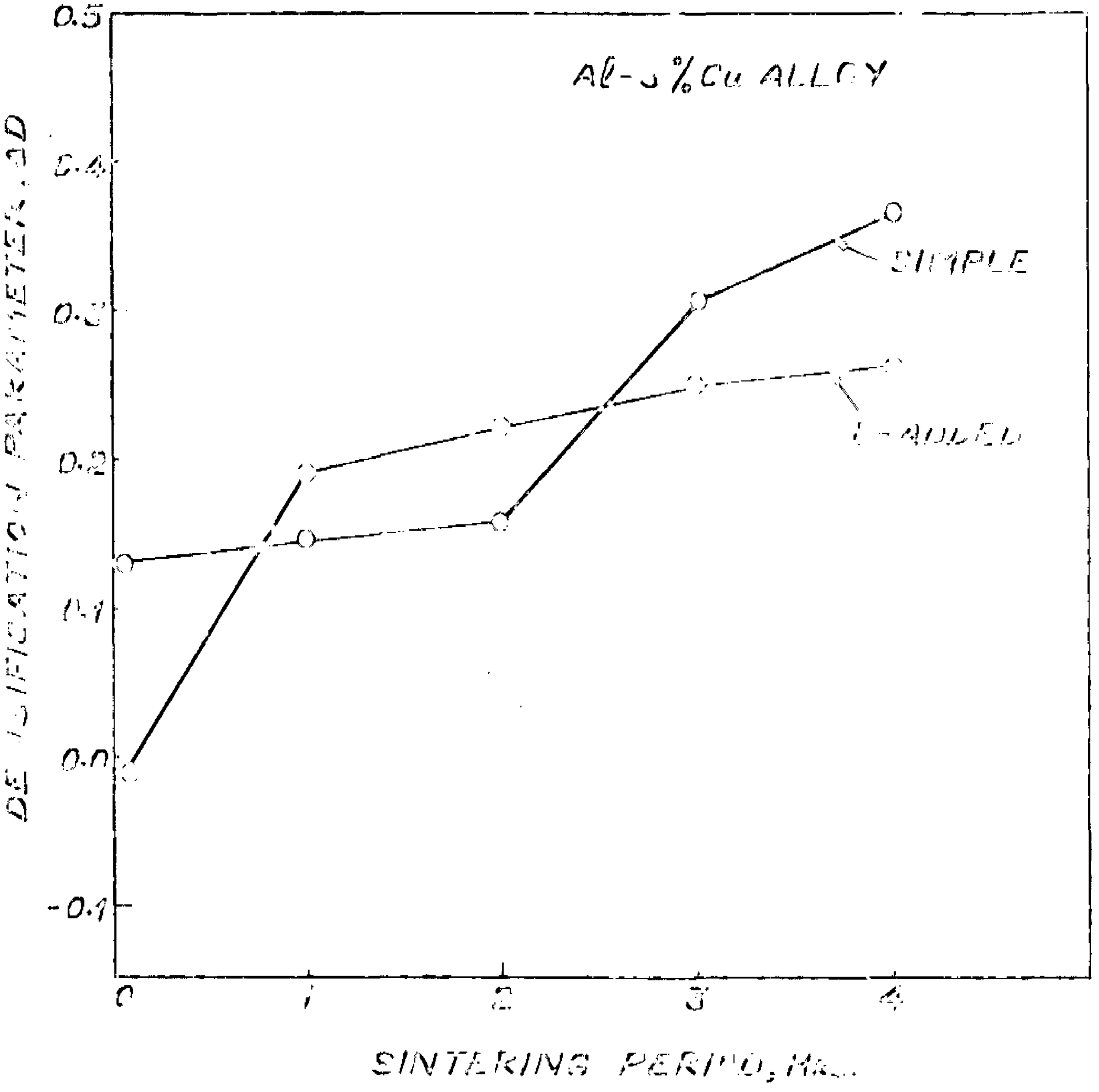


Fig.IV.5 [illegible] of [illegible]
[illegible] Al-5 Cu alloy (a) [illegible] (b) [illegible]
[illegible] at [illegible]; [illegible] temperature [illegible];
[illegible] pressure [illegible] tonnes/sq.cm.

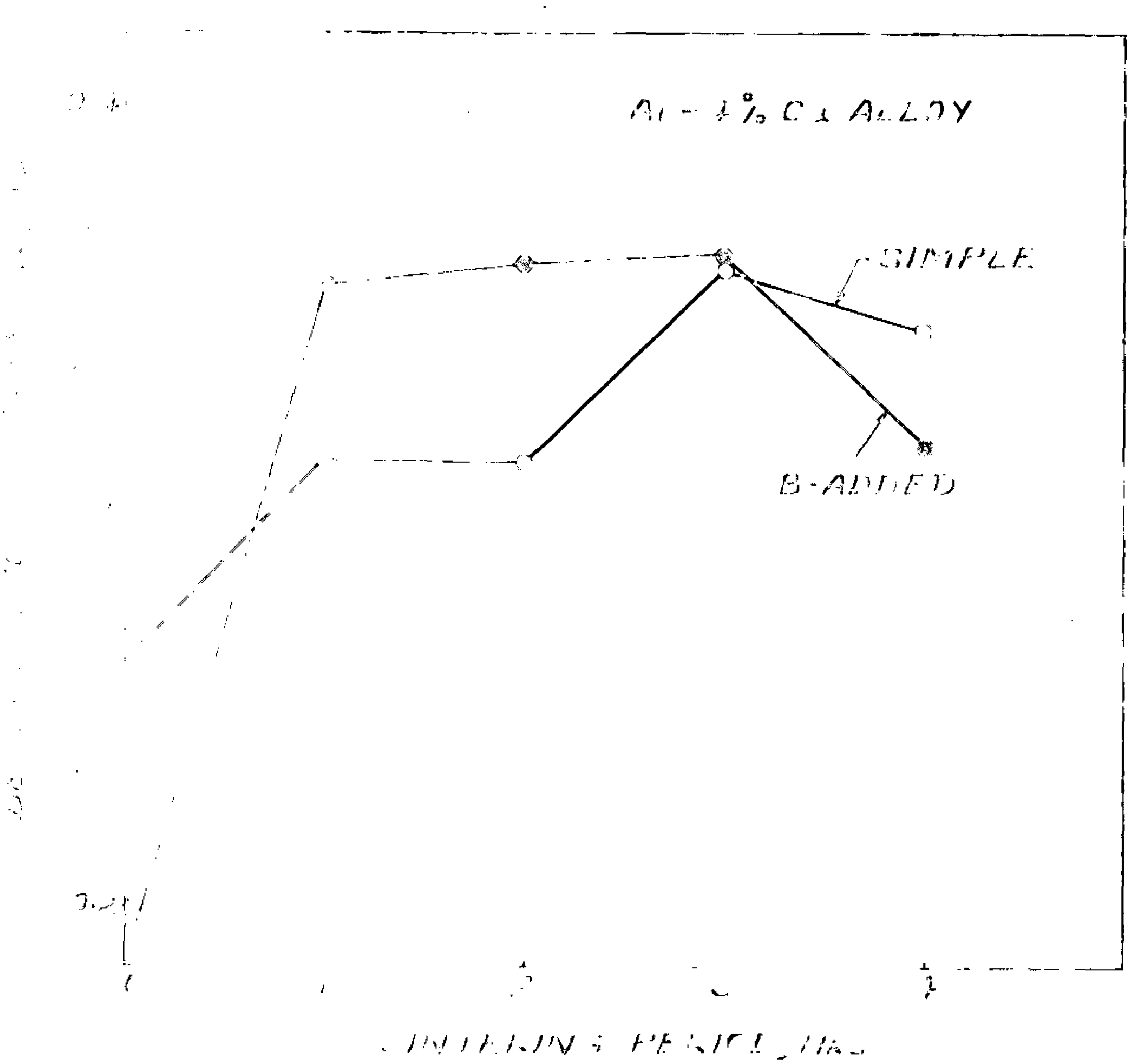


Fig. IV.6 Effect of Sintering Period on the Densification Parameters of Al-4% Cu alloy (a) Simple, (b) Boron added 0.05 wt.%, sintering temperature 500°C; Compacting pressure 2.5 tonnes/sq.cm.

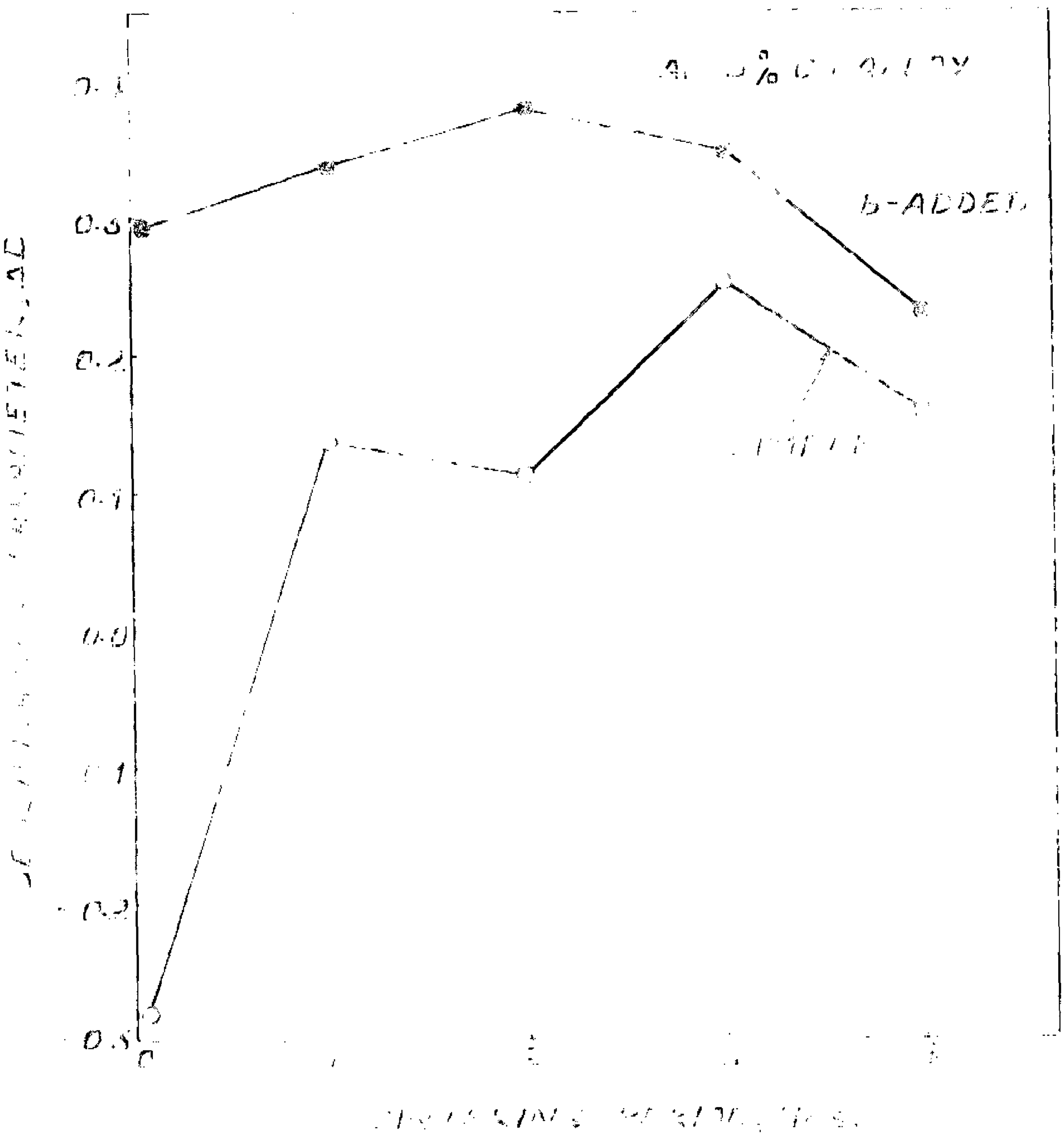


Fig. IV.7 Effect of sintering period on the densification parameters of Al-5% Cu alloy (a) simple, (b) Boron added (0.05 wt.%), sintering temperature 500°C; Compacting pressure 2.5 tonnes/sq.cm.

simple compacts when sintered for 5 minutes and then contraction with a maximum at 3 hours. In Al-5% Cu-.05% B compacts contraction gradually increases till a sintering period of 2 hours and decreases beyond this period. It is also observed that at any sintering period between 5 minutes and 4 hours Δ D values decrease as the Cu percentage is increased. This could not, however, be confirmed in case of Boron added samples.

Comparing the densification parameter values in Table IV.4 it becomes evident that the peak shifts towards lower sintering periods as the Cu content in Al increases, The ΔD values at peaks in Boron added specimens increase with increase in Cu content and lower sintering periods. This is, however, not clearly evident in case of simple Al, Al-Cu compacts.

IV.2.2 Hardness:

No marked change in hardness values of Al and Al-B compacts with change in sintering period is observed. In cases of alloy systems the Boron added specimens show significant changes in the hardness values as compared to simple ones.

Changes in hardness values for Al-3% Cu-B and Al-4% Cu-B are throughout positive i.e. till 4 hour sintering. But for Al-5% Cu-B the positive effect is obtained till a sintering period of 3 hours and is negative later. The peak values as pointed out in Table IV.5, on comparison illustrate that as the Copper content increases the peak shifts to lower sintering periods. It is further seen that maximum hardness (peak values) increases with

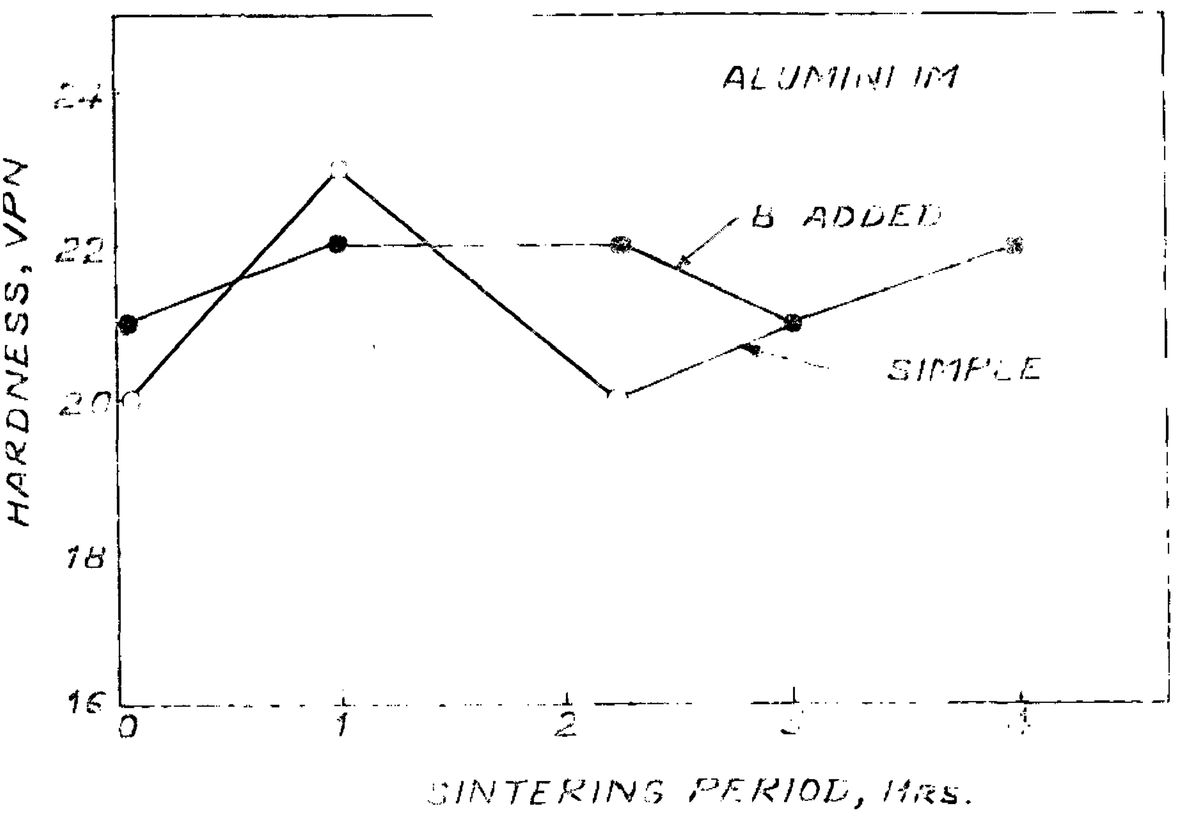


Fig. IV.8 Effect of Sintering Period on the Hardness Variation of Aluminium Powder Compact (a) Simple (b) Boron added (0.05 wt.%), sintering temperature 600°C; Compacting Pressure 2.5 tonnes/sq.cm.

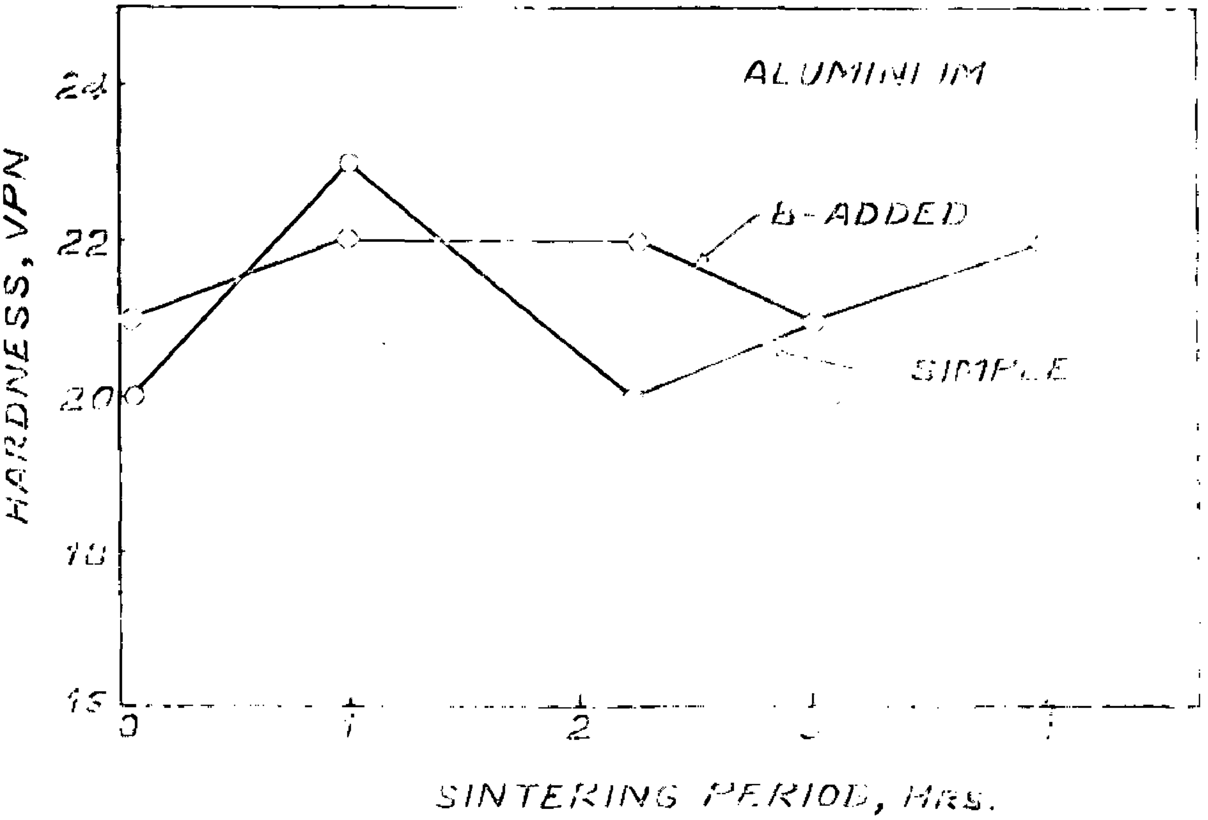


Fig. IV.[illegible] Effect of [illegible] period on the hardness variation of aluminium compact (a) simple (b) [illegible] added [illegible] [illegible] temperature [illegible]°; [illegible] [illegible] [illegible] tons/sq.cm.

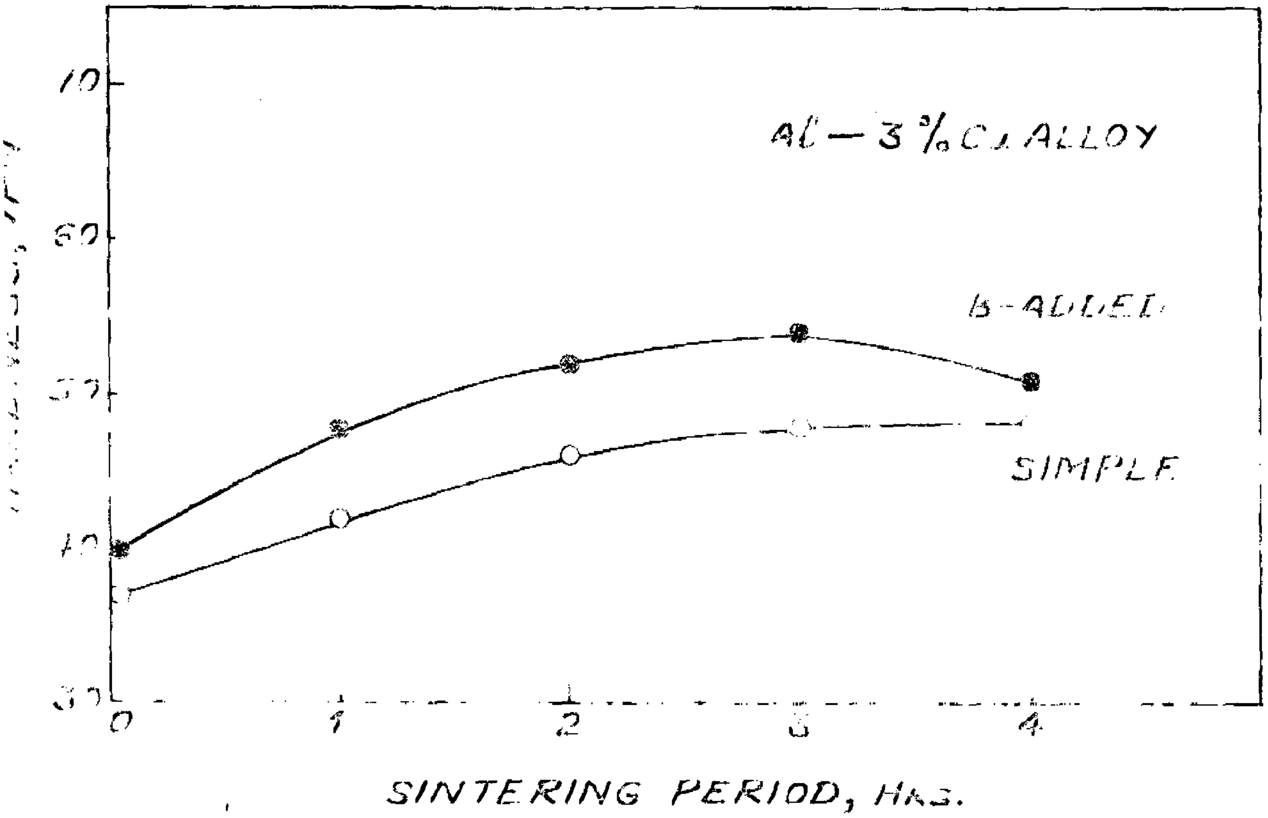


Fig. IV. 9 Effect of Sintering Period on the Hardness Variation of Al-3% Cu alloy (a) Simple (b) Boron added (.05 wt%); sintering temperature 600°C; Compacting pressure 2.5 tonnes/sq.cm.

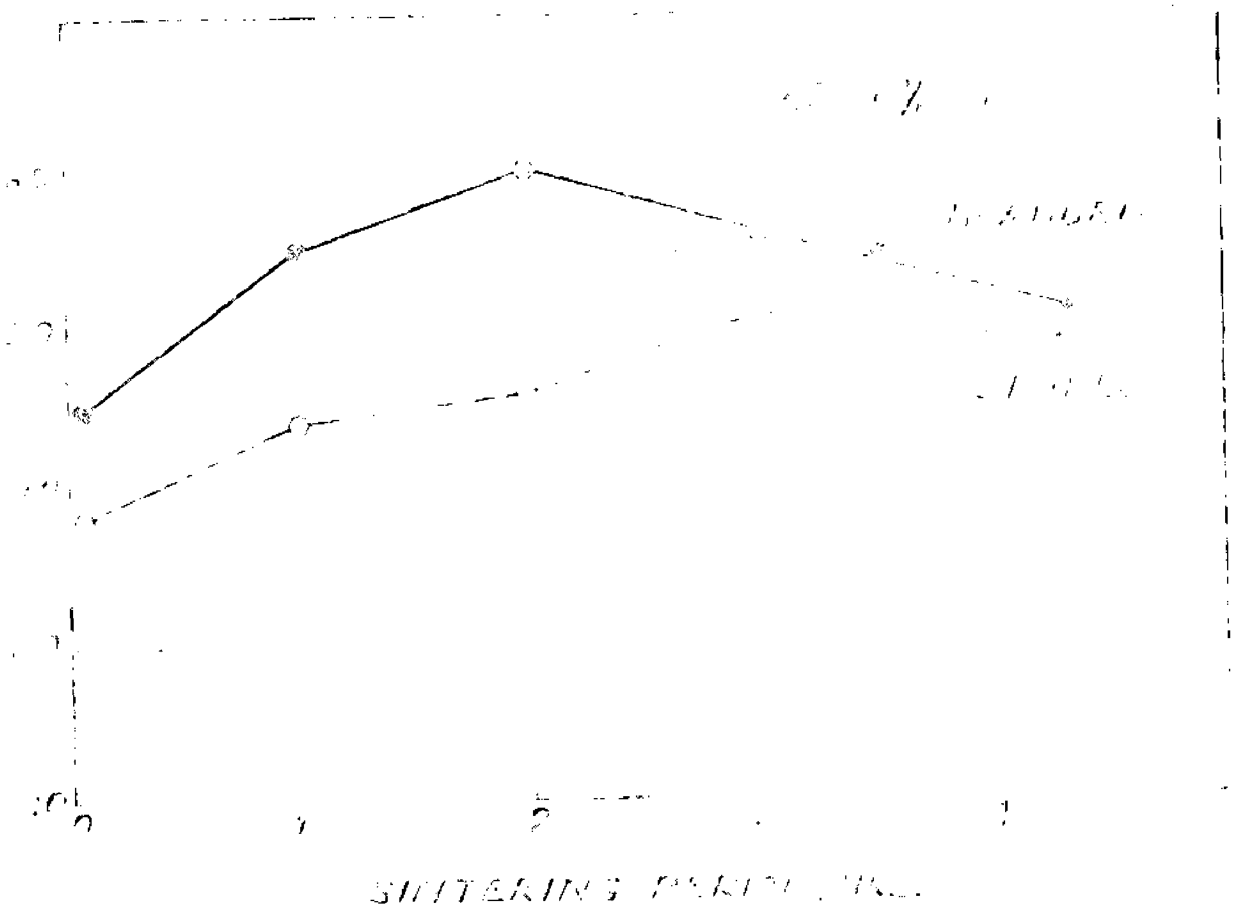


Fig. IV.10 Effect of Sintering Period on the Hardness Variation of Al-4% Cu alloy (a) Simple (b) Boron added (0.05 wt %); Sintering Temperature 600°C; Compacting Pressure 2.3 tonnes/sq.cm.

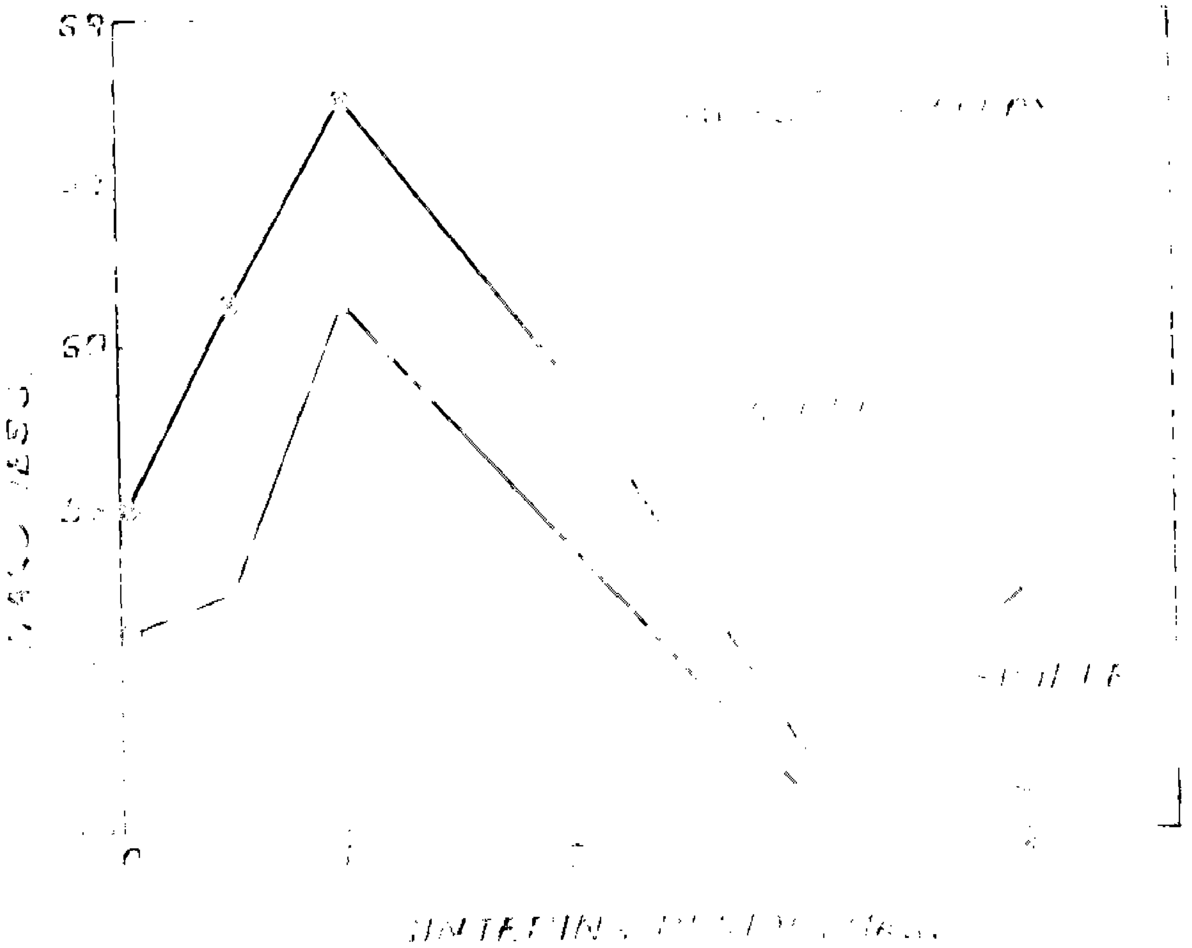


Fig. IV.11 Effect of Sintering Period on the Hardness Variation of Al-5% Cu alloy (a) Simple (b) Boron added (0.05 wt.%); Sintering Temperature 600°C; Compacting Pressure 2.3 tonnes/sq.cm.

TABLE IV.5 EFFECT OF COMPOSITION AND SINTERING PERIODS ON HARDNESS OF Al-Cu ALLOYS SINTERED AT 600°C

(a) SIMPLE

Alloy	5 mts.	1 hr.	2 hrs.	3 hrs.	4 hrs.
Al	20	23	20	21	22
Al - 3% Cu	37	42	46	48	48
Al - 4% Cu	38	44	46	51	49
Al - 5% Cu	53	61	55	49	54

(b) BORON ADDED

Alloy	5 mts.	1hr.	2 hrs.	3 hrs.	4 hrs.
Al	21	22	22	21	22
Al - 3% Cu	40	48	52	54	51
Al - 4% Cu	45	55	60	56	51
Al - 5% Cu	56	66	59	50	49

increase in Copper content in Aluminium in both activated and non-activated alloy mixtures. The above mentioned effect is much pronounced in case of activated ones, and is in consistence with the changes obtained in case of densification parameter values (Table IV.4).

IV.3 Microstructural Studies:

IV.3.1 Effect of Compacting Pressure on Microstructures of Aluminium Alloys:

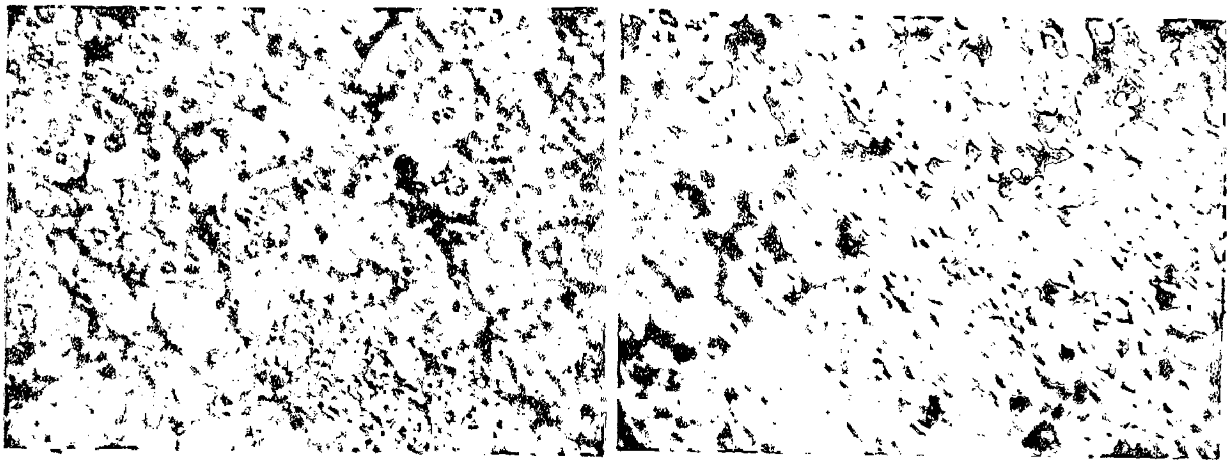
Microstructures of simple Aluminium and its alloys with 3%, 4%, 5% Copper corresponding to compacting pressures of 1.5, 2.3, 3.0 tonnes/sq.cm. respectively sintered at 600°C for 1 hour are shown in Fig.IV.12.

In simple Aluminium compacts porosity seems to interconnects as the pressure is increased. The Al-3% Cu compact pressed at 3 tonnes/sq.cm. is a compact structure and the pore size is small. This reveals maximum density as compared to other alloys pressed at the same (3 tonnes/sq.cm.) pressure. In microstructure of the Al-5% Cu alloy, compacted at 2.3 tonnes/sq.cm., the pores gather along the grain boundaries and is a homogenized structure. The pores spherodize when compacted at a pressure of 3 tonnes/sq.cm.

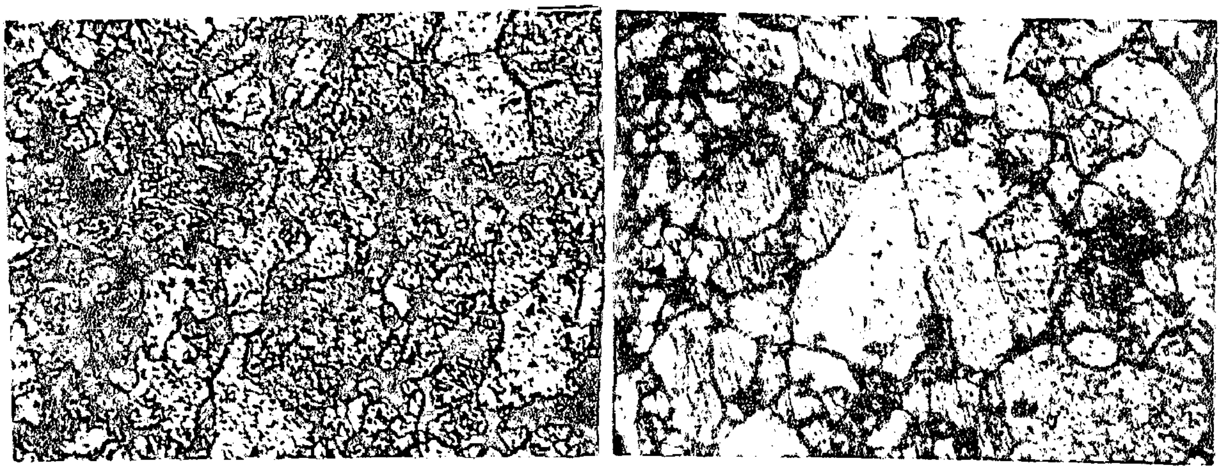
Microstructures of Al-3%, 4% and 5% Cu alloys reveal more porosity at a compacting pressure of 2.3 tonnes/sq.cm. than those compacted at other two pressures.

IV.3.2 Effect of Sintering Period on Microstructures of Aluminium Alloys:

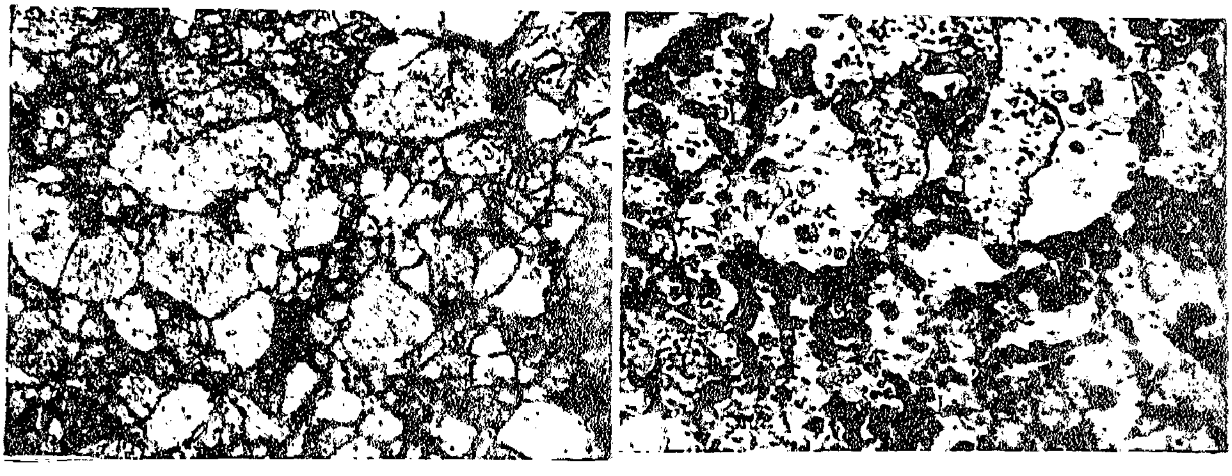
The microphotographs of simple and Boron added Aluminium



(a) ALUMINIUM POWDER COMPACT

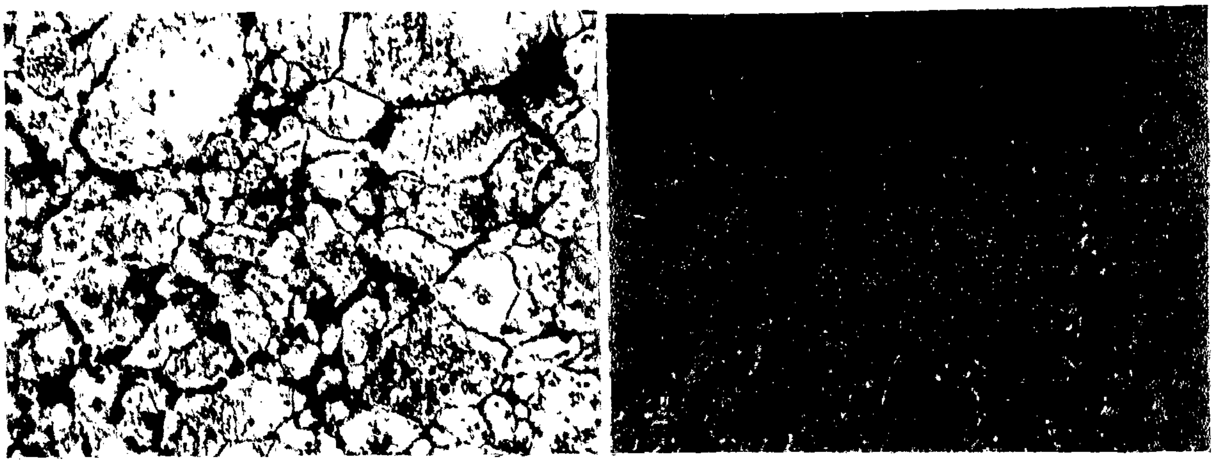


(b) ALUMINIUM 3% COPPER



(c) ALUMINIUM 4% COPPER

Contd.



(d) ALUMINIUM - 5% COPPER

FIG.IV.12 MICROSTRUCTURE OF ALUMINIUM AND Al-Cu ALLOYS COMPACTED AT 1.5 TONNES/CM^2 (LEFT) AND AT 3 TONNES/CM^2 (RIGHT); SINTERING TEMPERATURE 600°C; SINTERING PERIOD 1 HOUR compacts corresponding to a pressure of 2.3 tonnes/sq.cm. pressure, sintered at 600°C for various periods are shown in Fig. IV.13 (a,b,c,d,e). The microstructures of Al compact show some peculiar structures which change with increase in sintering period. These can not hence be correlated with experimental results. Boron addition has modified the structure at 1 and 2 hours, and increases the grain size at 3 and 4 hours.

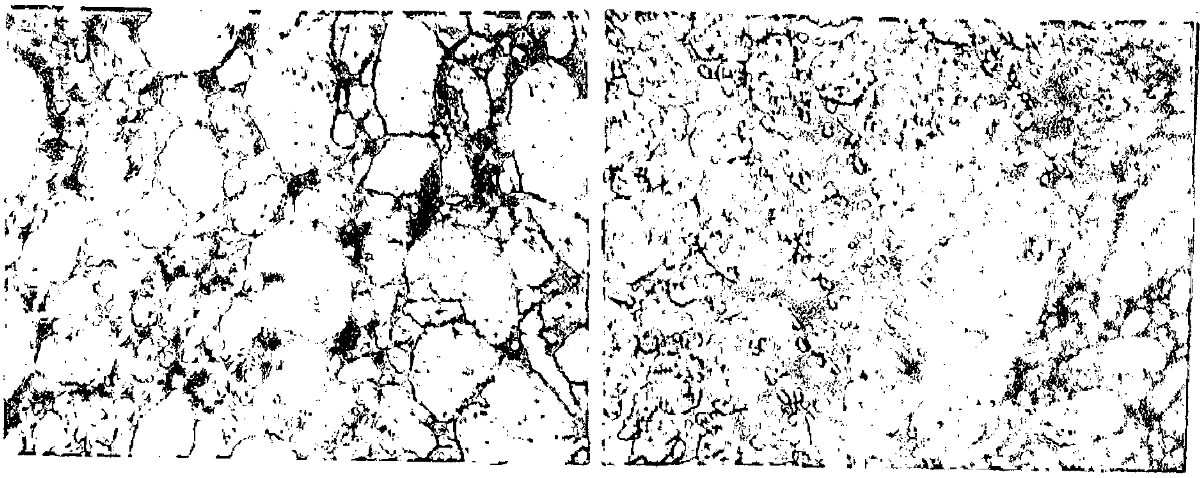
Microphotographs of simple and Boron added Al-3% Cu compacts are shown in Fig.IV.14.

The microstructures of simple Al-3% Cu compacts show densification with the increase in sintering period. The microstructures at 5 minutes show incomplete sintering. Homogenization seems to start at 3 hours of sintering. The alloying tendency is naturally more in case of compacts sintered for longer periods. Sintering period of 4 hours yields best microstructure among others obtained till 4 hours.

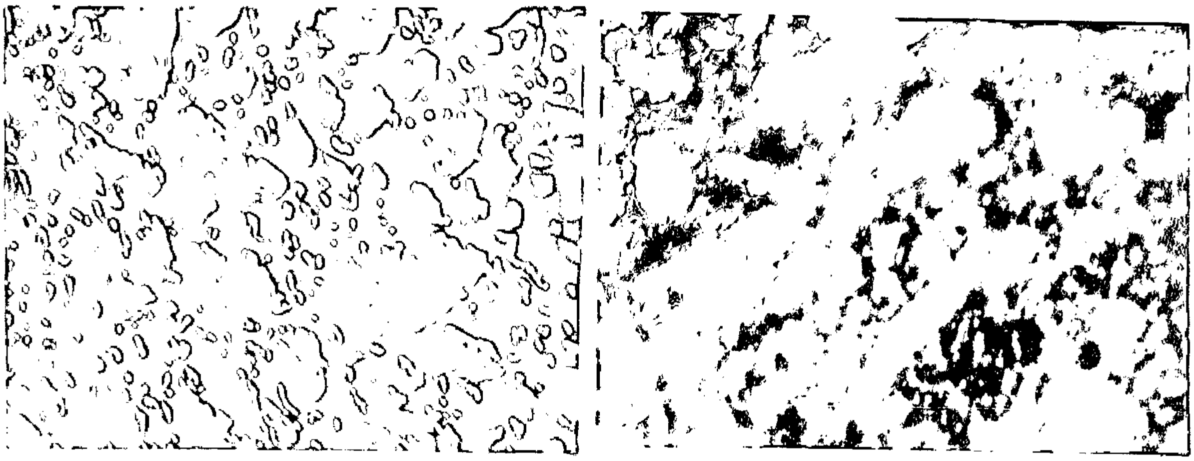
The Boron added samples shows densification in microstructures of compacts sintered for 1 and 2 hours, as compared to simple ones.

The microstructures of simple and Boron added Al-4% Cu alloy are shown in Fig.IV.15.

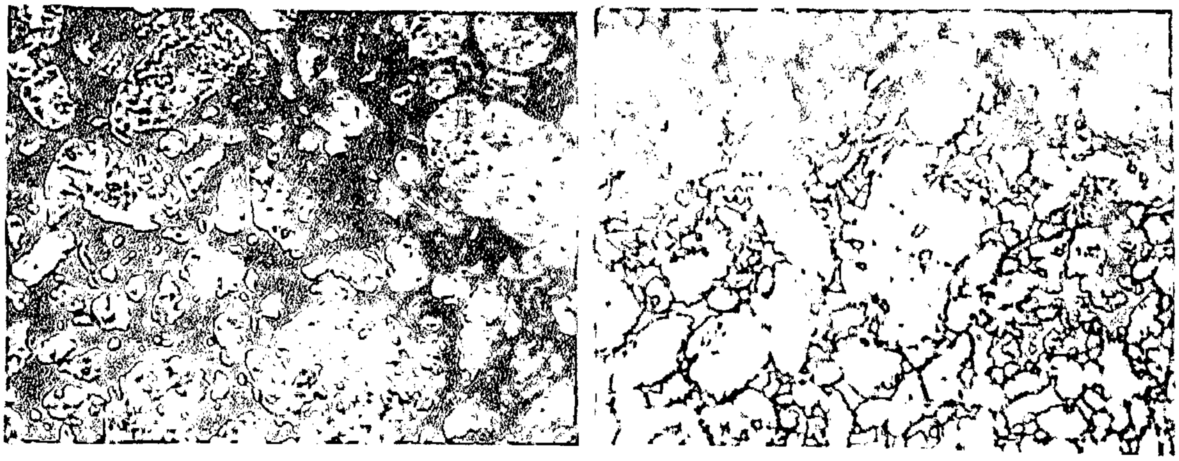
Simple Al-4% Cu structures densify with increase in sintering period. The decrease in ΔD value at a sintering period of 4 hour is not interpretable. The Boron activated structures can not be correlated with simple ones. More alloying of the



(a) SINTERING PERIOD 5 MINUTES

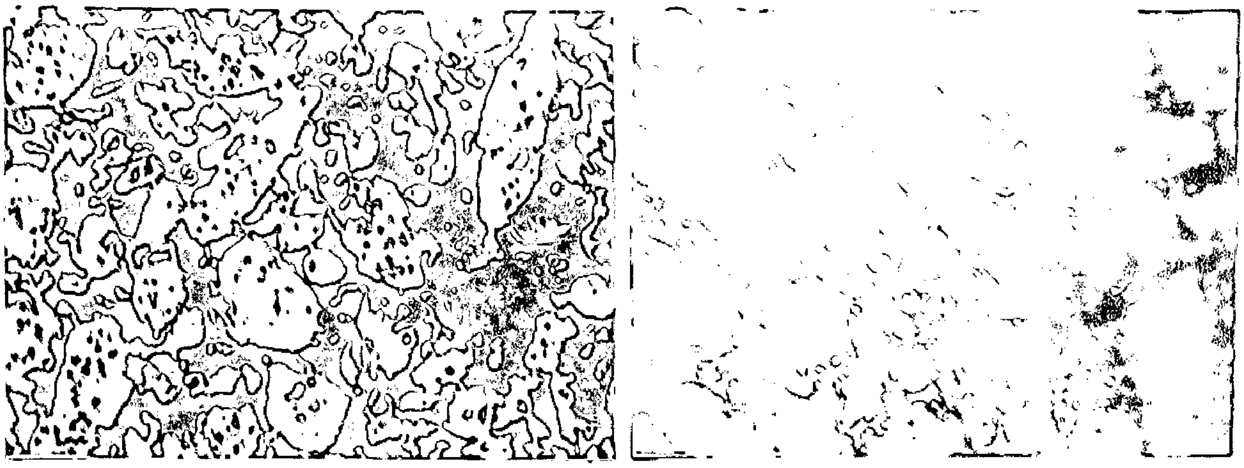


(b) SINTERING PERIOD 1 HOUR

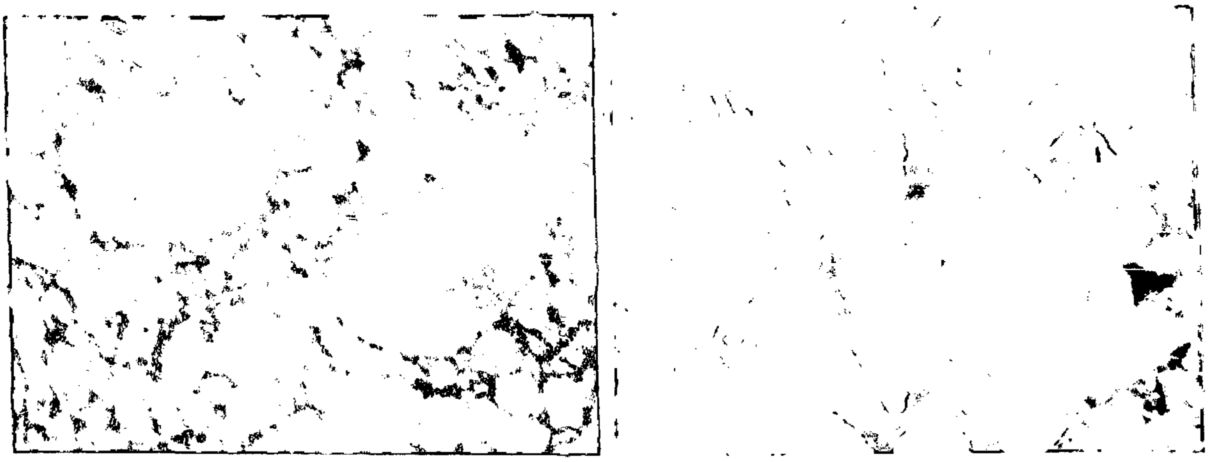


(c) SINTERING PERIOD 2 HOURS

Contd.

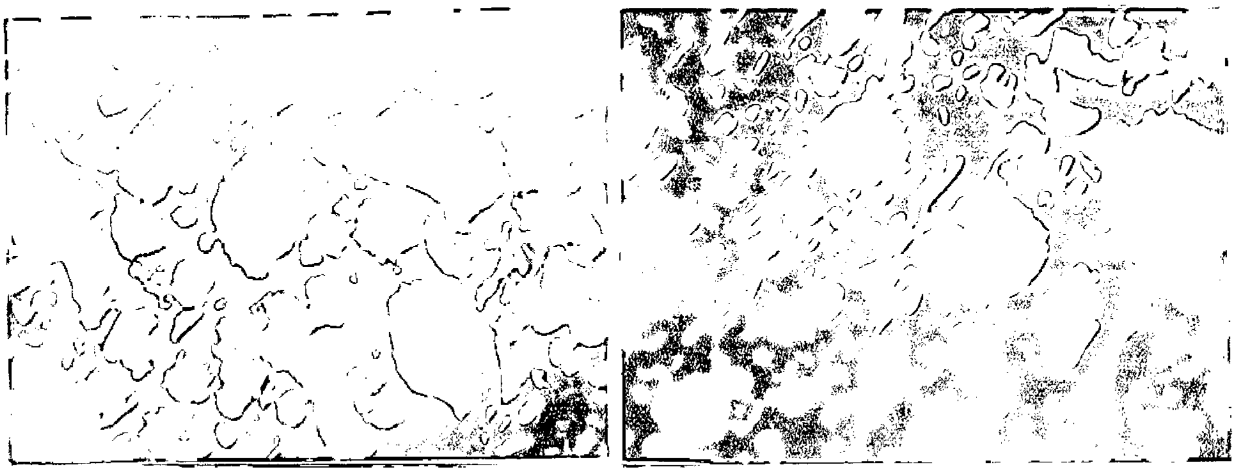


(d) SINTERING PERIOD 3 HOURS

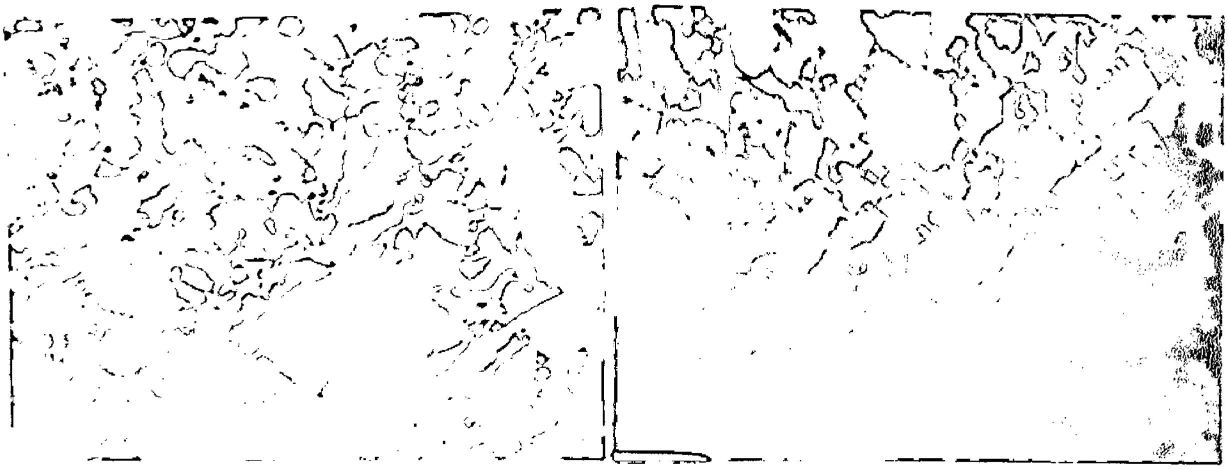


(e) SINTERING PERIOD 4 HOURS

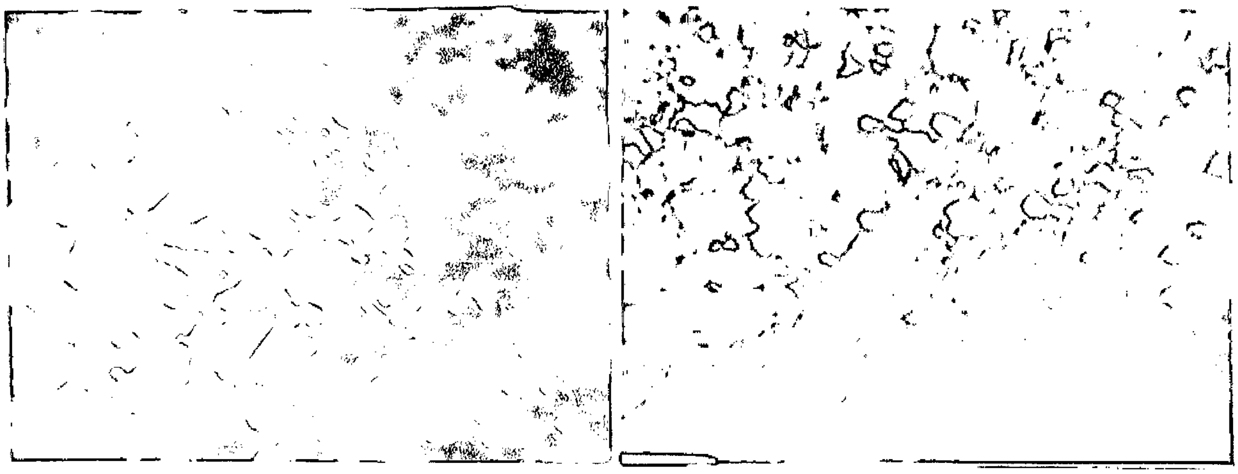
FIG. IV.13 MICROSTRUCTURES OF SIMPLE (LEFT) AND BORON ADDED (RIGHT) SINTERED ALUMINIUM COMPACTS, SINTERED AT 600°C FOR DIFFERENT SINTERING PERIODS; COMPACTING PRESSURE: 2.3 TONNES / SQ.CM.(X 150)



(a) SINTERING PERIOD 5 MINUTES

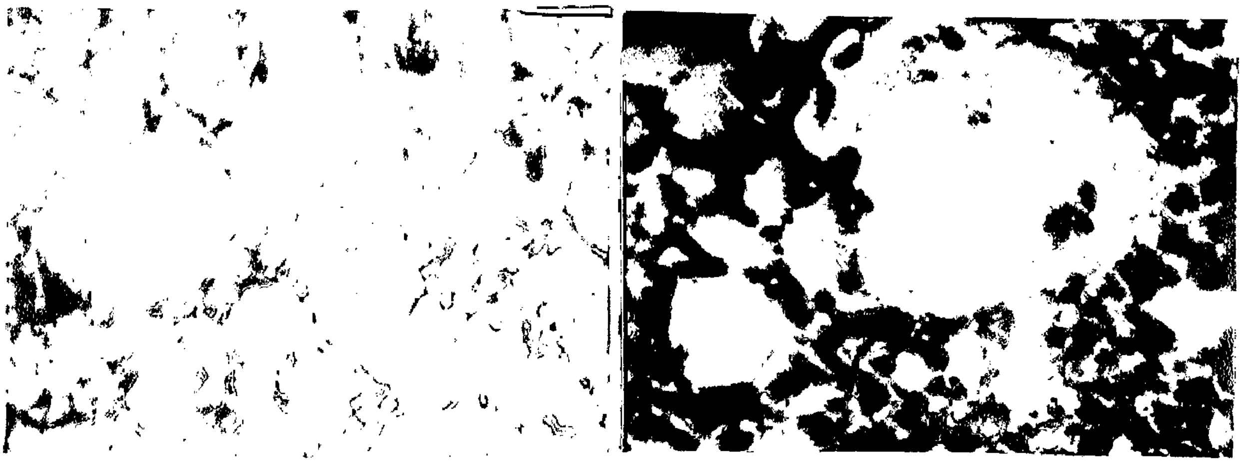


(b) SINTERING PERIOD 1 HOUR

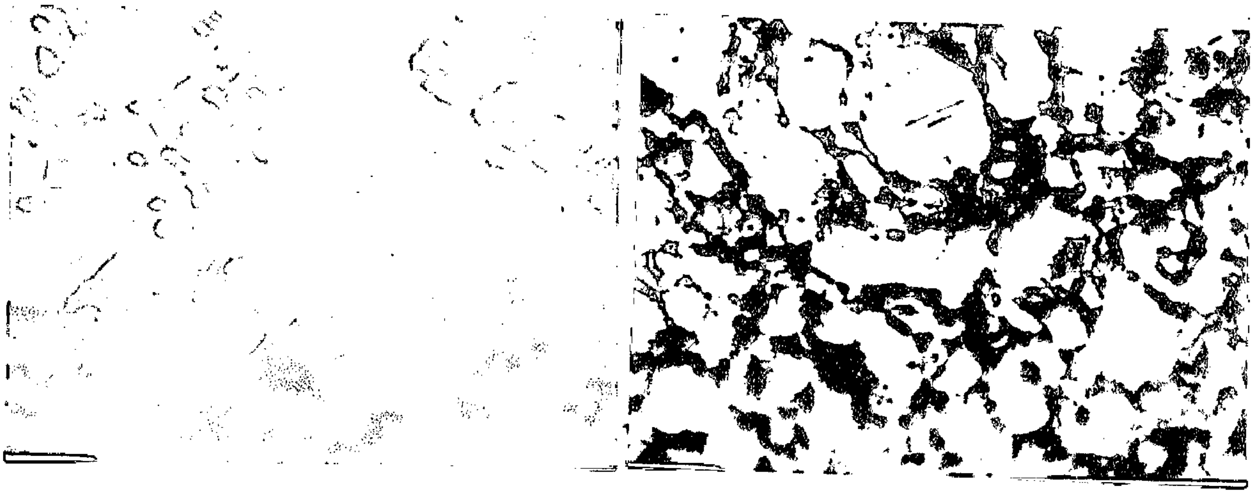


(c) SINTERING PERIOD 2 HOURS

Contd.

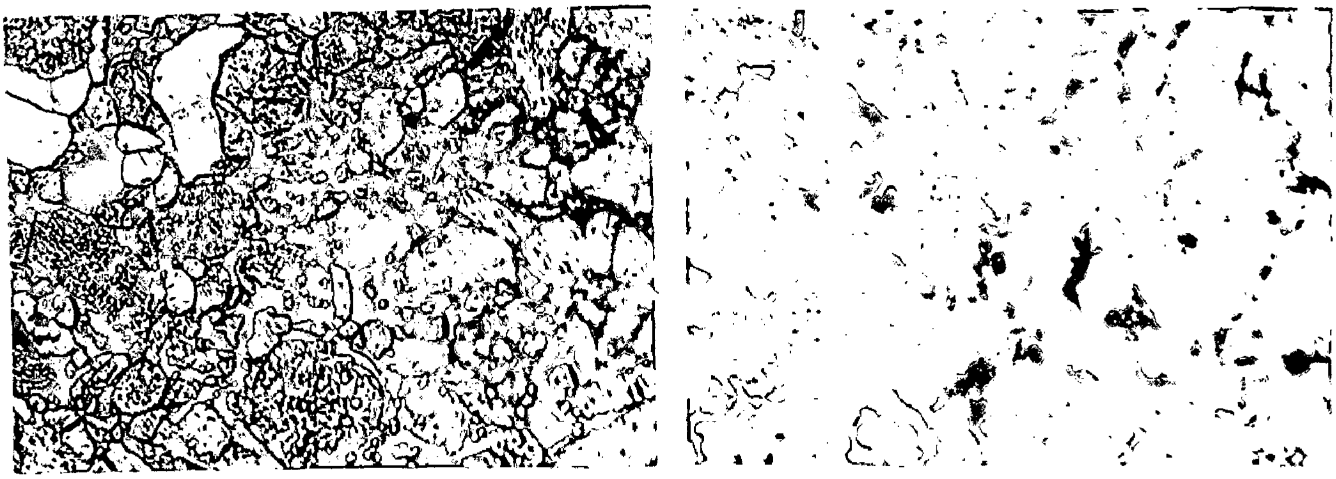


(d) SINTERING PERIOD 3 HOURS

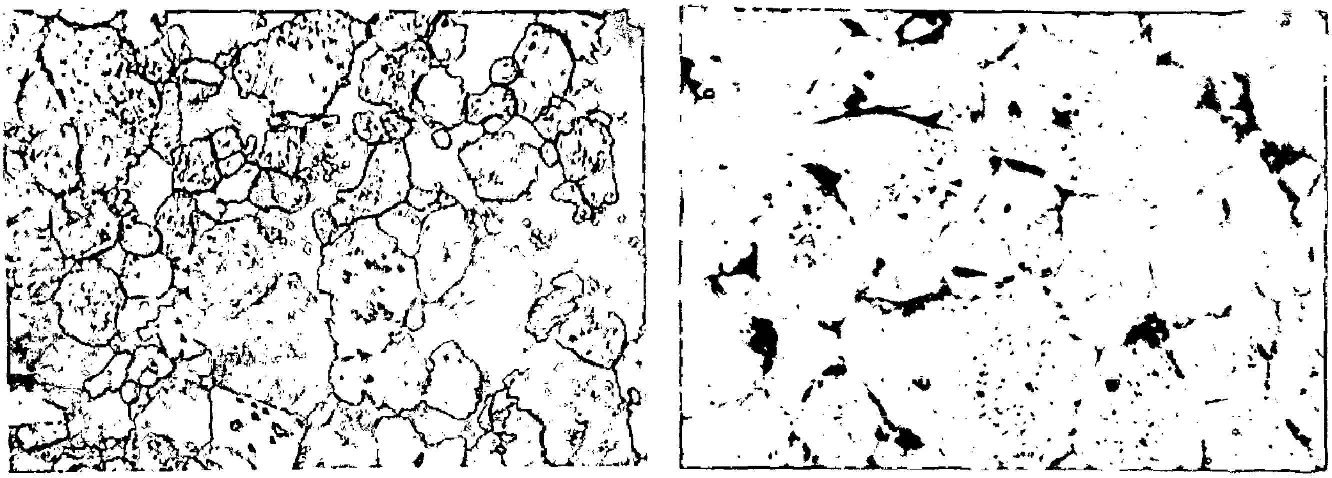


(e) SINTERING PERIOD 4 HOURS

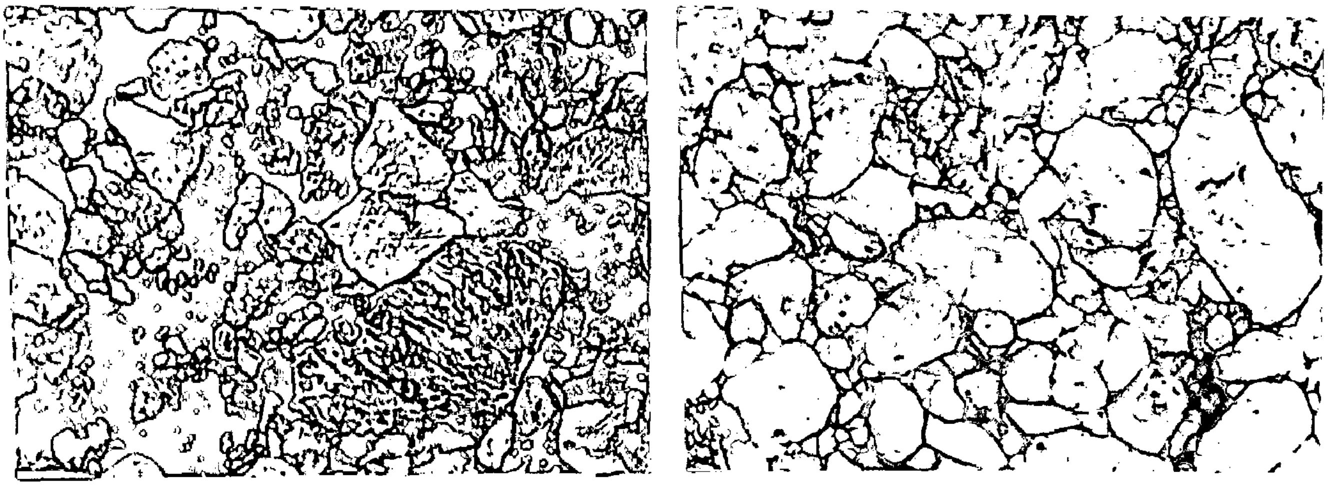
FIG. IV.14 MICROSTRUCTURES OF SIMPLE (LEFT) AND BORON ADDED (RIGHT) SINTERED Al-3% Cu COMPACTS SINTERED AT 600°C FOR DIFFERENT SINTERING PERIODS; COMPACTING PRESSURE 2.3 TONNES/ SQ.CM. (X 150)



(a) SINTERING PERIOD 5 MINUTES

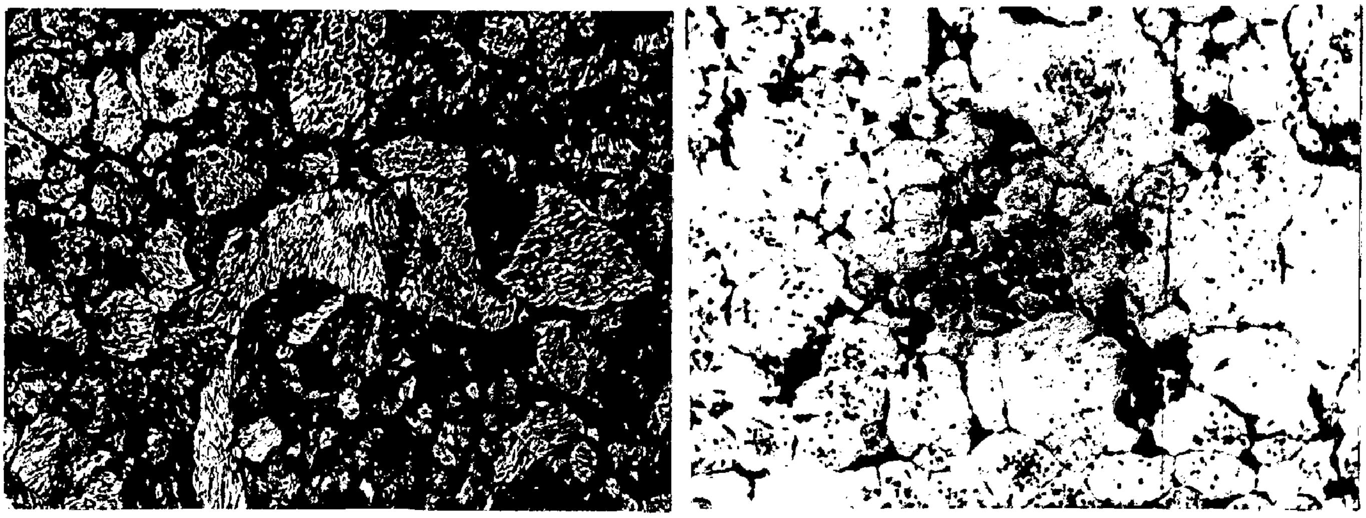


(b) SINTERING PERIOD 1 HOUR

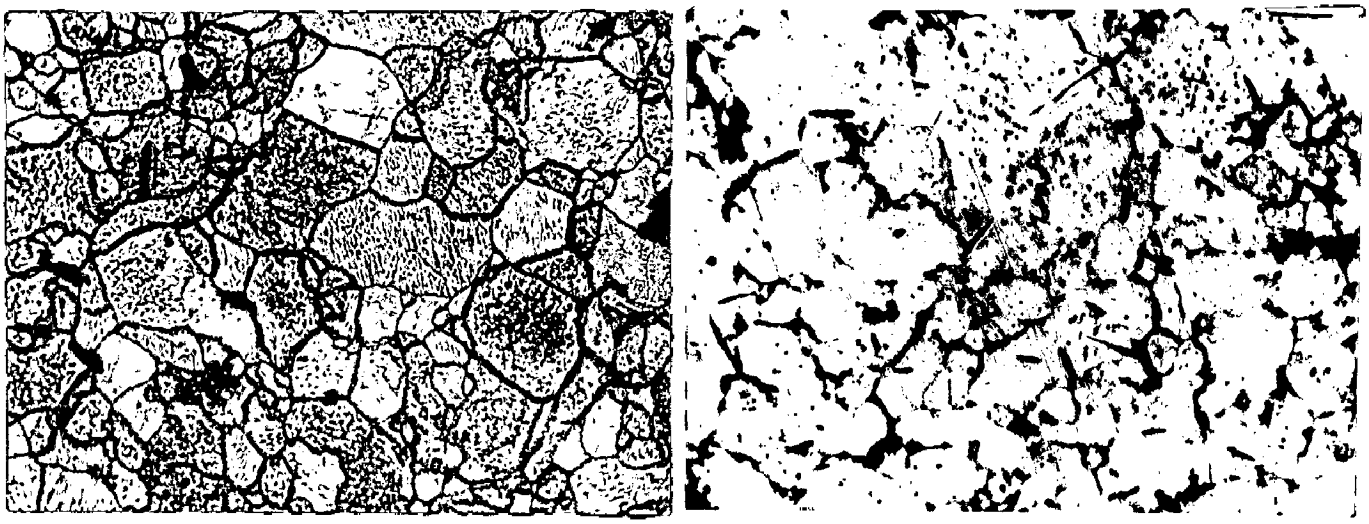


(c) SINTERING PERIOD 2 HOURS

Contd.



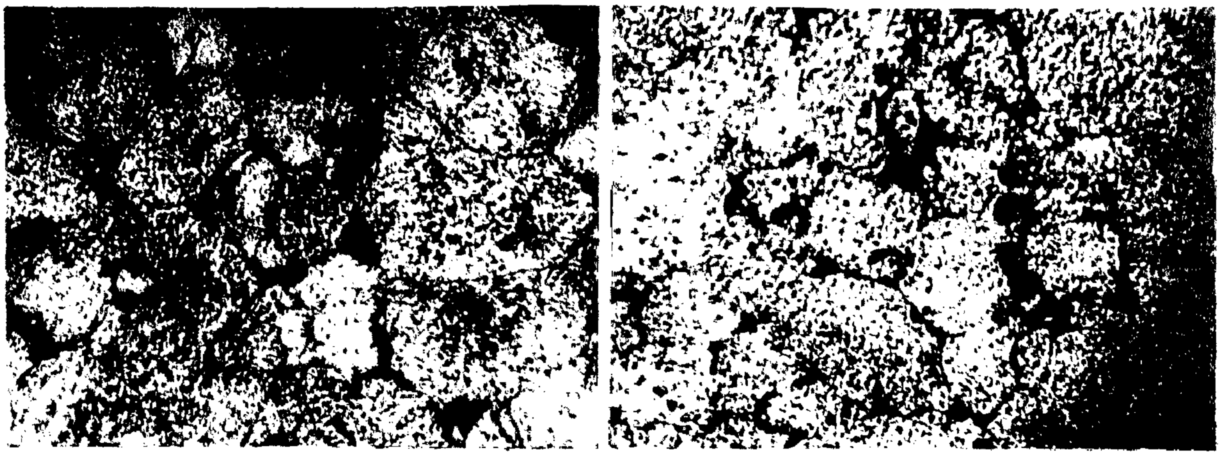
(d) SINTERING PERIOD 3 HOURS



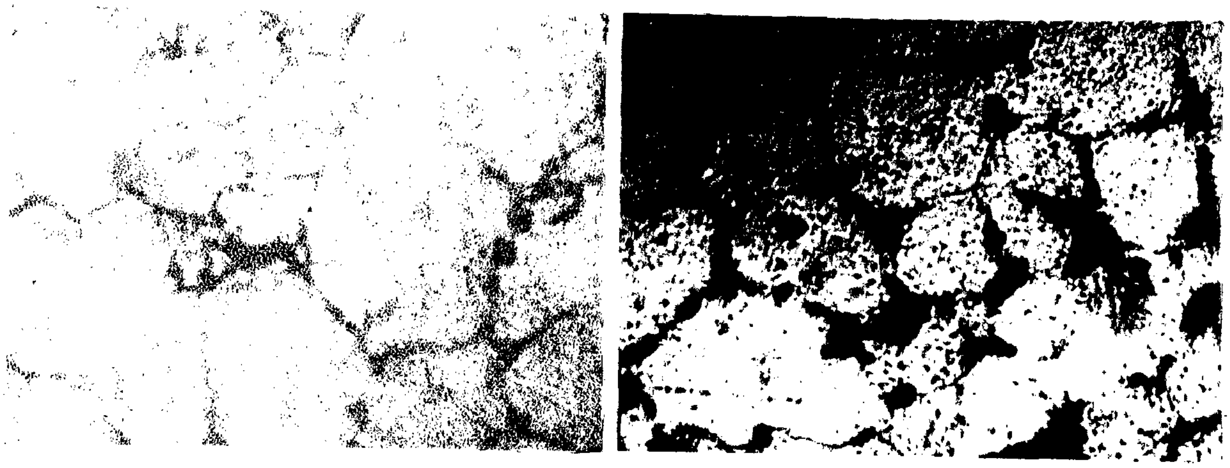
(e) SINTERING PERIOD 4 HOURS

FIG. IV.15 MICROSTRUCTURES OF SIMPLE (LEFT) AND BORON ADDED (RIGHT) SINTERED Al-4% Cu COMPACTS SINTERED AT 600°C FOR DIFFERENT SINTERING PERIODS; COMPACTING PRESSURE 2.3 TONNES/ SQ.CM. (X 150) structure is evident due to the change in etching behaviour of alloyed particles beyond 2 hours, which could be due to the presence of liquid phase.

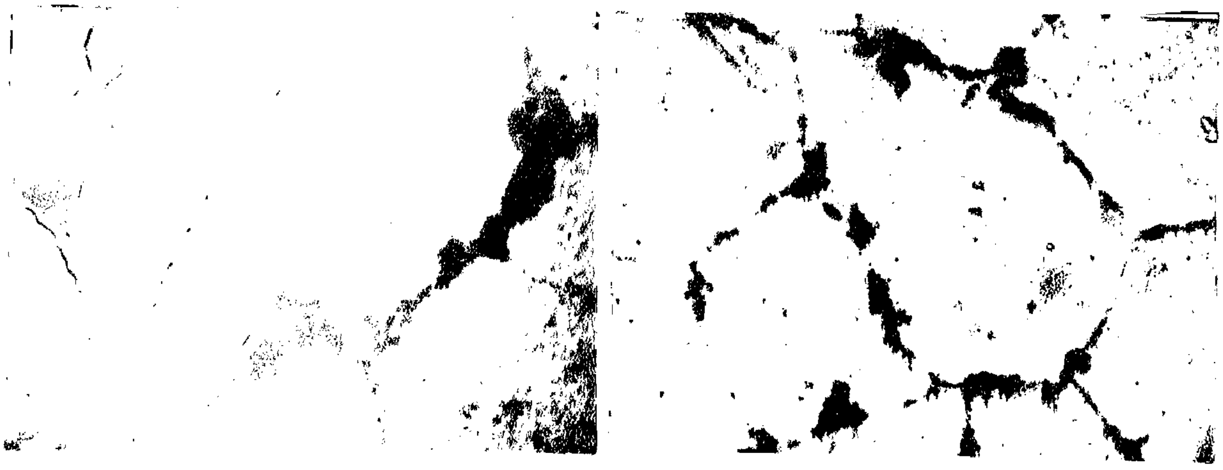
Microphotographs of simple and Boron added Al-5% Cu alloy are shown in Fig.IV.16. Although the pore size decreases with increase in sintering period till a sintering period of 3 hours, the increased grain growth can not be overlooked in this case. The homogenization is revealed by the difference in etching behaviour, Grain growth follows homogeneization. Grain growth is more in the case of Boron added compacts. It seems that grain growth commences only after sintering has proceeeded to a maximum extent, which corresponds to 2 hours.



(a) SINTERING PERIOD 5 MINUTES



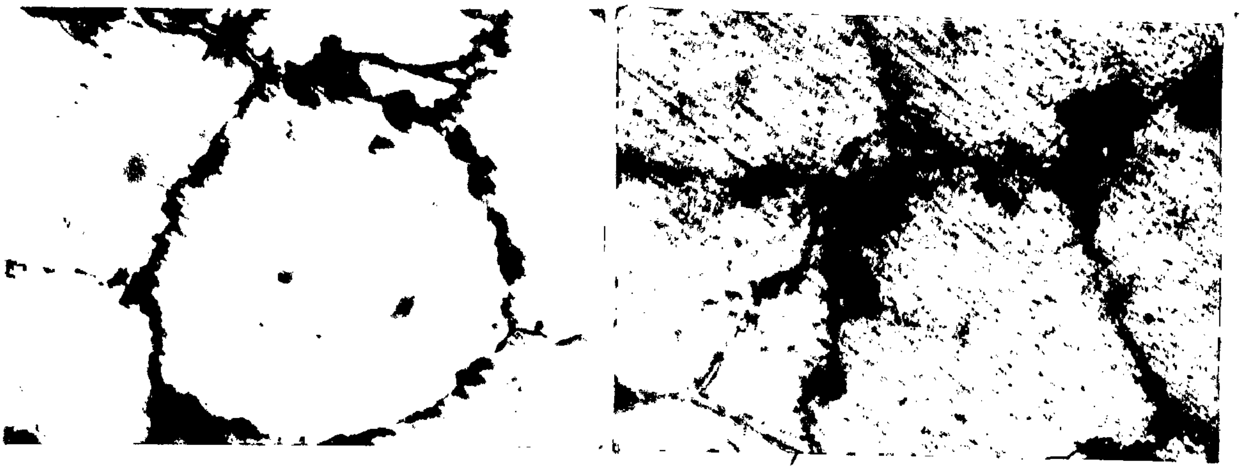
(b) SINTERING PERIOD 1 HOUR



(c) SINTERING PERIOD 2 HOURS

Contd.

(d) SINTERING PERIOD 3 HOURS



(e) SINTERING PERIOD 4 HOURS

FIG. IV.16 MICROSTRUCTURES OF SIMPLE(LEFT) AND BORON ADDED (RIGHT) SINTERED Al-5% Cu COMPACTS SINTERED AT 600°C FOR DIFFERENT SINTERING PERIODS; COMPACTING PRESSURE 2.3 TONNES/ SQ.CM.

CHAPTER V

DISCUSSION

V.1. Effect of Compacting Pressure on Sintering Behaviour of Al-Cu Alloy Compacts:

As the compacting pressure increases, porosity decreases till an optimum density is obtained. The densification is effected by the tendency of the particles to shift and reorientate themselves to the state of minimum energy, thus reducing the size and amount of pores. Both Al and Cu being FCC metals deform readily without getting appreciably work hardened. Accordingly only a low specific pressures will suffice to form green compacts from their powders. On raising the compacting pressure in case of Al-powder a uniform increase in green density is observed which becomes 93% of theoretical at a pressure of 3 tonnes/sq.cm. Sintering for one hour has improved the density of Al-compacts when compacted at 1.5 and 2.3 tonnes/sq.cm. respectively. This is verified by the microstructural studies (Fig.IV.12).

Thehardness of Al-sintered compacts increases with increase in compacting pressure in the initial stage, but it remains almost constant when the pressure increases beyond 2.3 tonnes/sq. cm. An increase in compacting pressure decreases the interparticle distance and thus increases the internal stress in the compact. This cases the brittle Al_2O_3film to break at some of contact points. The improved interparticle contact raises the hardness and other physical properties after sintering.

In Al-3% Cu alloy as the compaction progresses, more and more voids between the particles get filled up. This feature has been identical to the one observed by Martsunova et al (10). The densification parameter values of Al-3% Cu compacts give a minimum at a compacting pressure of 2.3 tonnes/sq.cm. which is in consistence with the microstructure (Fig.IV.12). The hardness variation also bears a similar plot and naturally interparticle strength improves with compaction.

Al-4% Cu alloy compacts show lower green densities than Al-3% Cu compacts. This may be attributed due to the bridge formation by copper particles at this composition which inhibits the complete flow of powder into the interstitials. The densification parameter however, shows a similar character as Al-3% Cu alloy, and achieves a better value when compressed at 3 tonnes/sq.cm. This could however, be not confirmed from the microstructural (Fig.IV.12). The hardness too shows a similar variation.

In Al-5% Cu alloy the green density increases, which is due to the increased Cu content in Al. The densification parameter values show a similar variation, the maximum ΔD value being at a compacting pressure of 3 tonnes/sq.cm., which decreases on further increase of pressure. This decrease in hardness may be due to the pore coarsening because of different gas pressure thus providing a driving force.

The densification parameter values of Al, Al-3%, 4%, 5% Cu at a compacting pressure of 3 tonnes/sq.cm. show opposite character as obtained in compacts when pressed at 1.5 tonnes/sq.cm the increase in ΔD value with respective increase in Cu content takes place, which is due to more alloying and hence more amount of equilibrium quantity of liquid phase. This fact is well established from the microstructures, as the porosity changes directly confirm with ΔD plots. The change in hardness values with composition also confirm the fact.

V.2 Effect of Sintering Period on Properties of Al-Cu admixed Powder Compacts:

V.2.1. Aluminium Powder Compacts:

Sintered aluminium compacts pressed at 2.3 tonnes/sq.cm. revealed contraction on sintering at 600°C (Fig.IV.8). The contraction increases with sintering period till one hour and beyond this period decreases. Densification parameter values show a positive effect of activation, till 2½ hours by Boron addition as compared to simple Al compacts. Beyond this period expansion is caused in Boron added Al compacts. Boron addition has delayed the achievement of densification in early stages of sintering, hence expansion is observed in the compacts sintered for 5 minutes. At such an early stage of sintering, it appears that Boron atoms are not interacted with the matrix and hence its particles are acting merely as dispersions then decreasing the ΔD value.

The structural and property changes show a rapid state of sintering. The sintering rate in simple Al compacts has proceeded at a decreasing rate after 1 hour Boron addition makes longer sintering periods possible, offering possibilities to achieve better combinations of hardness and densification parameter. The microstructures of simple and Boron added Al compacts do not show the densification behaviour clearly. The grain coarsening at 4 hours is evident (Fig.IV.13), which may be due to selective diffusion by a few grains which grow together giving rise to increased hardness.

Aluminium sintering at 600°C does not form any liquid phase and the densification is achieved due to enhanced rate of diffusion by the activation of Boron in solid state. The hardness variation of sintered Al compacts does not clarify the sintering behaviour. Boron added Al compacts show negligible effect of sintering period on hardness.

V.2.2 Aluminium -3% Copper Alloy:

The effect of sintering period has not been pronounced on the ΔD values till 2 hours. Longer sintering periods increase the densification rates which is attributed due to the presence of liquid phase formed at longer sintering periods. These are in consistence with the microstructures (Fig.IV.14). Although in case of Al-3% Cu alloy solidus falls at 600°C in Al-Cu equilibrium diagram, yet this alloy appears to be more akin to the formation of liquid phase. This is evidenced by the nature of densification and hardness curves (Fig.IV.5 and 9), which relate to those of higher Cu containing alloys. Hence a small amount of liquid phase ($<$ 1%) must have formed incipiently during sintering.

The effect of Boron addition on densification in this alloy is positive at sintering periods of 1 and 2 hours, is confirmed by the microstructures also. The ΔD values of Boron added compacts increase uniformly with increase in sintering period but it has again delayed the achievement of densification, caused due to non-uniform packing of particles and non-uniform size distribution. The hardness values of the simple Al-3% Cu compacts increase with sintering period and give a maximum at 3 hours. Here Boron addition increases the diffusion rates of compacts which result in increased hardness values.

V.2.3 Aluminium - 4% Copper Alloys:

The density of simple Al-4% Cu alloy increases with sintering period upto 3 hours and there is a decrease beyond that, which is not explained by the microstructures. Boron addition once again delayed the sintering rate, which is attributed to the same reasons already mentioned in the cases of Al and Al-3% Cu sintered compacts.

Boron addition increases the ΔD values, the effect of which is positive till 3 hours of sintering. Densification in simple and activated Al-4% Cu alloys is confirmed by the microstructures. The decrease in density of Boron added compacts beyond 3 hours is due to the redistribution of segregated porosity. In the present alloy liquid phase to the extent of 5.6%

could form at the sintering temperature 600°C under equilibirum condition. However, since we are not reaching equilibrium, the real amount of liquid would be less than this value. The formation of liquid phase activates the sintering of compacts by increasing the density because of increased flow of liquid. It could also be concluded from Fig.II.5, that the liquid phase helps in scattering the oxide films and facilitates the formation of better contacts. (9)

Boron addition may form low melting ternary phases increasing the amount of liquid phase, resulting in increased wetting and hence increased density. Although Boron addition in Al reduces the liqudus temperature of Al (Fig.V.1) yet due to non-availability of sufficient literature in ternary diagrams for Al-Cu-B the above mentioned effect could not confirmed.

The hardness values of this particular alloy increase with sintering periods till 3 hours. Boron added compacts give much higher hardness values and at the same time reduce the sintering period essential to achieve the maximum possiblehardness offering a still better combination of Δ D and hardness values (Fig.IV.6 and 10). Higher hardness of sintered compacts as compared to Al-3% Cu is attributed to the solid solution hardening as well better sintering because of larger proportion of liquid phase present.

V.2.4 Aluminium - 5% Copper Alloys:

The densification parameter values of this alloy are much affected by Boron addition, the maximum being obtained at 2 hours.

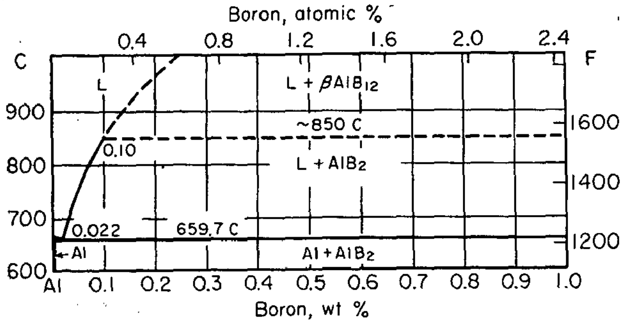


FIG. V.1

ALUMINIUM-BORON BINARY PHASE DIAGRAM. (24)

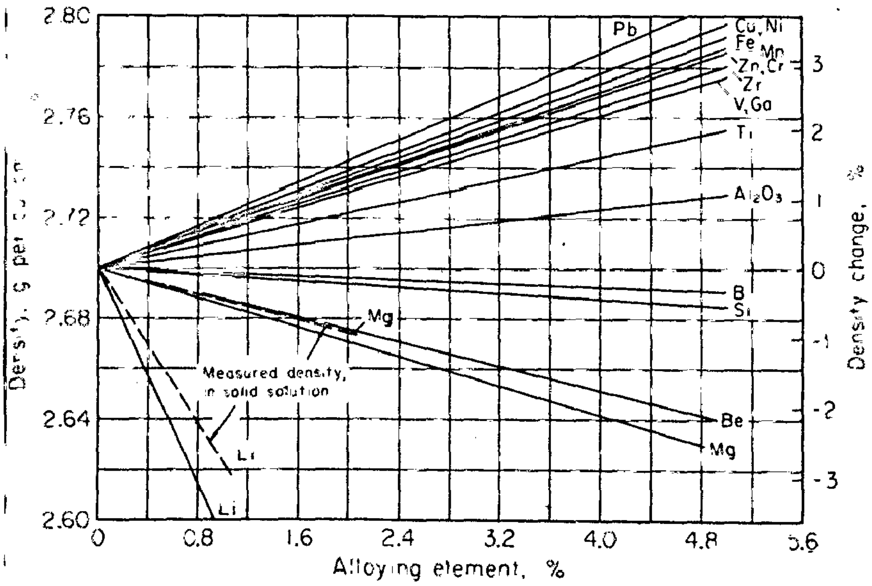


FIG. V.2

EFFECT OF COMPOSITION OF VARIOUS ELEMENTS ON DENSITY OF ALUMINIUM (24)

Boron addition has reduced the sintering period by one hour to obtain the maximum. The D results correspond to the microstructures one after 1 hour of sintering. It is important to note that the delayed densification observed.

Densification observed in Al, Al-3%, 4% Cu alloys at an early stage of sintering caused by the dispersion of Boron is eliminated in this case due to the formation of a sufficient amount of liquid phase (10.6%), which forms at a comparatively low temperature as solidus falls to 560^{o}C (Fig.II.10).

The saturation of hardness in Al-5% Cu alloy is attained at a relatively short period i.e. 1 hour.This is also due to relatively longer time available for the liquid phase to diffuse into the matrix because of the above mentioned factors. The decrease in hardness beyond one hour sintering appears to be related to grain growth as evidenced in the microstructure (Fig. IV.16). The densification of structure takes place by close approaches of the individual crystallites, that takes place as sintering progresses. Hence the liquid phase introduction during sintering is an expedient which accelerates the achievement of properties. The boundaries of the crystallite have a deciding influence on the resulting properties of the sintered metal. As the original particle boundaries are weaker, the recrystallization associated with grain growth results in better properties of the material than is possible in unrecrystallized compacts. The formation of a closely point structure is attributed by surface tension forces. Once a homogeneous solid solution is formed, further metallographic changes are only coarsening of the structure. The larger grains of Al-Cu and Al-Cu-B alloys may have formed due to the interparticle alloying. During cooling of the alloy, the liquid phase present at that temperature solidifies and is deposited into remaining Al-Cu crystallites resulting in rather large sized alloy particles in the final structure.

For any composition of alloy investigated presently the hardness value increases with the increase in alloying content and hence with sintering period. Comparing Figs. IV.9,10, and 11 the Cu content increases, the maximum hardness is obtained at a lower sintering period which is due to more rapid alloying. Boron addition activates the sintering process, as it gets dissolved upto an extent of .001% in Al (24).

Table IV.4 shows the effect of composition and sintering period on densification. In Boron added specimens, ΔD values increase with composition, but respective maxima shift towards lower sintering periods. This does not relate well with the ΔD values of simple compacts. However, hardness values in Table IV.5 show a similar change in peak values with composition for both simple and Boron added alloys.

Thus it is obvious from the above discussion that the activation effect of Boron on sintering is more felt in high copper content Aluminium alloys.

Recent work of Naidich(26) has shown that the surface tension of liquid Al, with alloying by Cu increases with concentraction. This change is rather rapid one after 10% of Cu in the alloy. In the present investigation the concentration of Cu in liquid will be more in higher Cu content alloys. This suggests that wettability will be poor in initial stage of liquid phase formation (at temperatures just above solidus) in higher Cu containing alloys. This would naturally effect the liquid flow stage during sintering such that in high Cu content alloys, although the amount of liquid is more, densification parameter is slightly lower than the lower Cu containing alloys. Since such a feature is not observed in Boron added specimens one can be certain to select high Cu containing alloys with Boron to achieve better overall properties of densification as well as hardness. We can therefore conclude that while discussing the liquid phase sintering aspect of admixed metal powders the stages of alloying and its related physical effects must be deeply foreseen.

CHAPTER VI

CONCLUSIONS

1. Isothermal sintering of Aluminium and Al-3%, 4%, 5% Cu alloys was carried out at 600°C in vacuum of order of 10^{-4} mm of Hg.

2. 0.05% Boron addition was made in the above alloys and it was observed that Boron activates the sintering process.

3. Green compacts of above systems were prepared at different compacting pressures viz. 1.5, 2.3 and 3 tonnes/sq.cm. It was observed that with increase in pressure the green density increases and in case of alloys the density approximately increases with the alloying content.

4. Densification parameter variation, of above alloys sintered at 600°C for 1 hour, with respect to compacting pressure exhibits different patterns for Aluminium, and Al-Cu alloys such that in case of former ΔD decreases whereas in latter cases it increases with compacting pressure. From the plots, the optimum compacting pressure for the alloys was found as 2.3 tonnes/sq.cm. where the ΔD variation was minimum. Hardness variation of above mentioned alloys bear a similar nature except for Al, where hardness increases even with loss of ΔD.

5. Densification parameter variation of Aluminium - 3%, 4%, 5% copper alloys with respect to sintering period, when sintered at 600°C shows the presence of peak such that with increase in alloying addition, the peak shifts towards the lesser sintering periods. This feature is more reflected in case of Boron-activated sintered compacts.

6. A similar plot variation is observed in case of hardness variation of sintered compacts with respect to the sintering period. The hardness peak increases in magnitude when alloying addition is more and is observed at a lesser sintering period. However, Boron addition does not show improvement of Δ D and hardness in all the cases uniformly. The microstructural observations confirm the above relationships.

7. The results have been interpretted on the basis of different propertions of liquid phase in different Al-Cu alloys, such that larger the amount better the sintering because of the mass transport mechanism due to liquid flow. The activation effect of Boron appears to decrease the liquidus temperature of Aluminium as well as the binary alloys thus enhancing the liquid phase sintering.

REFERENCES

1. SW McGee, SAE, Paper No. 710531, Vol.80, 1971, pp.1719.
2. C.G.Goetzel : Treatize on P/M, Vol.I, Interscience Publishers, 1950, pp.489.
3. C.G. Goetzel : Treatize on P/M, Vol.II, Interscience Publishers, 1950, pp.489.
4. C.G. Goetzel : Treatize on P/M, Vol. II, Interscience Publishers, 1950, pp. 723.
5. Alcoa Staff Report : Metal Progress, Vol. 99, No.4, 1971, pp. 60.
6. Kempf L.W. : Powder Metallurgy, edited by J. Wulff, Am. Soc. Metals Cleveland, 1942, pp. 314.
7. Cremer and Cardiano : Trans. Am. Inst. Mining Met. Engrs. 1943, pp. 152.
8. Bickerdike : Symposium on P/M. The Iron and Steel Institute, Special Report No. 38, London, 1947, p.185.
9. Wantanabe and Yamada : International Journal of P/M, Vol.4, No.3, July 1968.
10. Martsunova et al : Poroshkova Mett. No. 12, 1973, pp.14.
11. F.J. Esper and G. Lenze : Powder Met International, Vol.3, No.3, August 1971, pp. 1-3.

12. Nachfrgal : Proc. International P/M Conf., Graz. July 12-17, 1948, Referate No. 30.

13. J.H. Dudas and W.A. Dean : Progress in P/M, MPIF, Vol. 25, 1969.

14. Paul E. Mathews : International Journal of PM, Vol.4, No.4 (59) 1969.

15. J.H. Dudas and C.B. Thompson : Modern Developments in P/M, Vol. 5, pp.19.

16. Material Engineering, Sept. 1973, Vol. 78, No.3, pp.27.

17. Material Engineering, Sept. 1973, Vol.78, No.3, pp. 18.

18. Metal Progress April 1971, Vol. 99, No.4, pp. 81.

19. Ramakrishna and Tendolkar : Powder Met. International, Vol. I, No.2, 1969.

20. G.P. Halbfinger : Int. Journal of P/M, 8(4) 1972, pp.231.

21. P.V. Thareja : Term Paper, Deptt. of Met. University of Roorkee, Roorkee, May 1973 (Unpublished work)

22. 'Market Break Through of Al Sinters' Light Metal and Metal Industry, Sept. 1965, pp.35.

23. Kent R. Van Hom : Aluminium, Vol. I, ASTM, Nov. 71, pp.168.

24. L. M. Parker, and U. Colombo : The Science of Materials used in Advanced Technology, John Wiley and Sons, Inc., 1973, pp.118.

25. L.R. Parker, U. Colombo : The Science of Materials used in advanced Technology, John Wiley and Sons, Inc., 1973, pp. 118.

26. Yu. V. Naidich, Kontaktnie Yulenia V. Metallicheskikh Rasplavakh., Naukova Dumka, Kiev, 1972, p.155.