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Precipitation Hardening of Al Mg Si Alloy Reinforced by SiC Particles

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ABSTRACT-:
The material produced in this investigation was 6061 aluminium alloy with 5-20 wt% SiC particulates. The unreinforced matrix alloy with an identical thermal history has also been studied in order to establish a basis for comparison. It was found that the overall, age-hardening sequence of the alloy, was not changed by the addition of SiC particles The aging behaviour of unreinforced and reinforced materials were studied in both natural and artificial aging at 170°C. The results show that the incorporation of 5 to 20wt.% SiC particles can, however, be considerably improved by natural or artificial aging. The reaction kinetics of the precipitation process has been accelerated by the introduction of reinforcement On the other hand, the peak aged hardness of these composite materials is comparable with or even less than, that of the unreinforced alloy. Also, the effects of deformation for both unreinforced and composite alloys were studied. The results show that the deformation altered the aging precipitation sequence especially when aging at 170°C, the more deformation, the higher dislocation densities and hence, the faster the precipitation. Generally, deformation accelerated aging and hence, peak hardness occurred earlier. Appreciable decrease in hardness and faster kinetics were obtained by introduction of thermomechanical processing to these alloys.

Key words-:
Metal matrix composite, precipitation hardening, microstructure, aging, silicon carbide particles, heterogeneous nucleation, deformation, thermomechanical treatment.

INTRODUCTLON:-

The physical and mechanical properties that can be obtained with aluminium based metal matrix composites (MMCs) have made them attractive materials for aerospace and automotive applications[1,2]. More recently, particle reinforced metal matrix composite have attracted considerable attention as a result of their relatively low costs and isotropic properties.

When a particular reinforcement is introduced into an heat-treatable aluminium alloy matrix, similar aging characteristics might be expected for both composite and unreinforced material. However, in practice these composites do not always realize their potential strengths and can even exhibit impaired strengthening when compared with the unreinforced alloy. The kinetics of aging can also be different and both enhanced and retarded behaviour has been exhibited in different MMC systems[3-5]. An accelerated aging phenomenon is reported in the 6061 Al-B_4C particulate composite in comparison with the unreinforced matrix alloy. This acceleration is attributed to a decrease in the incubation time required for the nucleation event and a concomitant increase in the solute diffusivity and, therefore, in the rate of precipitate growth[6,7]. Also, experiments on Al-Cu, Al-Cu-Mg, and Al-Mg-Si composites have shown that age hardening may be substantially decreased by using an average size of $\cong 1\mu m$, and, in some composites, there is no age hardening but rather may be age softening[8,9].

These various trends are not consistent but, nevertheless, the general observation in most particulate reinforced metal matrix composites that the aging kinetics are enhanced because of particulate reinforcement is usually attributed to an increase in the matrix dislocations density arising from the mismatch in the values of the coefficient of thermal expansion (CTE) between the matrix and the reinforcements[10,11].

The.present work was undertaken to address these inconsistencies. So, the main objective of this research is, therefore, to examine the effect of SiC particulates on the aging behaviour of 6061 aluminium matrix composite. Also, the influence of deformation and preaging singly or in combination are studied on the aging kinetics of matrix and composite alloys.

EXPERIMENTAL:-

The matrix material used in the present study was 6061 aluminium alloy containing (in wt.%), 0.6Si -1.0Mg -0.35Fe -0.25Cu -0.25Cr -Al (bal). Silicon carbide particulates with an average size of 40μm were selected as the reinforcement phase. Four casting experiments were made to synthesize unreinforced alloy and composites containing 5, 10, and 20wt.% SiC particulates. The composites were produced using stirring casting technique by dispersing SiC particles in molten 6061 Al-alloy. The details of this method was explained in a previous work[12]. For the purpose of comparison, separate ingot of the base alloy was also produced following the similar casting parameters.

Aging studies were carried out on specimens sectioned from matrix alloy and composites in order to find out the time required to attain peak hardness. It essentially involved solutionizing of sample for 2 hrs at 540°C, quenching in cold water followed by aging for various intervals of time 1- aging at room temperature, 2- artificial aging at 170°C, 3- 10 and 30% cold deformation after solution treatment, followed by natural and artificial aging, and 4- TMT consists of preaging at room temperature for 24 hrs., then cold deformation by 10, 20, 30 % and then final aging at 170°C.

All the prepared specimens were immediately refrigerated when not in use to prevent room temperature aging. Hardness measurements were performed using a Vickers hardness tester with a load of 10 Kgf. Each hardness value was averaged over eight measurements. Hardness values were determined as a function of aging time for different aging heat treatment conditions.

RESULTS:-

Microstructure:-

The microstructures of the unreinforced and reinforced 6061 Al-alloy in the cast condition are shown in Fig. 1 . The matrix structures of composites show smaller grain size than of 6061 Al-alloy. At the same time, the increasing percent of SiCp, on the microstructure showed more fine grain size. These results may be due to the presence of SiCp which act as sites of nucleation during solidification of the melt. Microstructure of the composite alloys show regions of clustered SiC usually associated with some porosity. Such clusters develop during solidification, then size of clusters being dependent on the rate of solidification, faster solidification results in smaller clusters[13]. The increase in weight percent of SiCp resulted in the formation of agglomerated particles. These particles tend to agglomerate in small groups; therefore, the particle spacing varies locally.

Some intermetallic inclusions of different phases like AlFeSi of Chinese script expected to exist in both matrix and composite alloys[13]. They are somewhat larger in the composite than the matrix alloy.

It is apparent that an interfacial reaction has occurred there by leading to the formation of spinel ($MgAl_2O_4$) at the particle/matrix interface[14]. Detailed inspection has shown that this interaction layer is irregular in thickness but not wider than 1 μm[13]. These interfacial reactions are important because they affect the composition of the matrix alloy and thus serve to influence the age hardening characteristics.

Also, Fig 2 shows the grain size distribution of both unreinforced and reinforced 6061 Al-alloy with different wt.% of SiCp. The grain sizes of these alloys were measured by interdendrite arm spacing. Generally, it was observed that the grain size of the matrix alloy exhibited a wide range of sizes measured in terms of the largest dimensions. This scattering of grain size was smaller in the composite alloys.

Aging behaviour:-

Fig. 3 shows the variation of matrix hardness as a function of aging time at room temperature for both unreinforced and reinforced alloys. The general shape of the aging curves for both materials show similar characteristics, which means that the addition of SiC in 6061 Al-alloy does not change the aging sequence. During natural aging, a smooth gradual increase of hardness is observed in all the materials to a certain level and then it levels off. The maximum increase of hardness values for the composites and the 6061 Al-alloy are fairly similar in magnitude and lie within the range of 64-68 HV. Although, there is scatter in the individual data points at peak aging, there is no evidence for any systematic difference in the values of hardness between composite and matrix alloys. At the same time, the results show that the time to reach peak hardness is less in the composite alloys than in the unreinforced alloy. This time was ranged between 112-76 hrs in composite alloys, while it was 128 hrs in matrix alloy.

Also, Fig. 4 shows a comparison of aging behaviour at 170°C for both composite alloys and 6061 Al-alloy. A closer examination of these curves show that the hardness increases initially with increasing aging time up to a maximum, after which it decreases with further increase in aging time. This means that the unreinforced and reinforced alloys show similar aging response. However, the time to reach peak hardness is less in the composite material(12-17 hrs) than in the unreinforced alloy (19 hrs). But, the reverse is the case of the maximum obtainable hardness. The degree of hardening attained via aging time is reduced in the composites in comparison with the unreinforced Al-6061 matrix Fig(4).

Generally, the differences in the peak hardness values between the unreinforced and reinforced alloys are small compared to the reduction in the peak aging time of the composite materials.

The detailed results from these aging curves are contained in Table (1). In this table, the aging kinetics has been summarized in terms of the aging time to peak hardness.

Effect of deformation:-

Hardness-time curves of naturally aged alloys with different degrees of deformation for unreinforced and reinforced alloys are shown in Figs. 5, 6. The general curve of these alloys with different degrees of deformation follows the same aging sequence as without deformation, i.e., peak hardness value has not been attained. A small variation of peak hardness values ranged between 63 HV for 6061 Al-alloy and 56-62 HV for composite alloys resulted from different degrees of deformation. On the other hand, deformation-accelerated aging and hence the maximum hardness values occurred earlier. It can also be seen that the greater the amount of SiCp present, the more rapid is the acceleration of the aging process, Table 1.

Also, Figs. 7 and 8 show the change in hardness with the aging time at 170°C with 10, and 30 % deformation for the unreinforced and composite alloys. The results show that the deformation altered the aging precipitation sequence. In the presence of cold work, there is a drop in hardness at the initial stage of aging for both 10 and 30 % deformation. The value of which is greater for 30 % cold work than in the case of 10 % cold work. On continued aging, the hardness shows a maximum in the aging curves followed by a reduction in the hardness with further increase in aging time. Also, the results show that the peak-aged hardness was higher in the unreinforced alloy than in the composite alloys at different degree of deformation and different wt.% of SiCp, Table 1.

Similarly, as naturally aged alloys, the aging time required to attain peak hardness is influenced by the amount of deformation and wt.% of SiCp and hence the maximum hardness values occurred earlier. The results revealed that aging time for peak hardness taken with 10% deformation was 12 hrs for the 5 wt.% SiC, 11hrs for 10 wt.% SiC, and 10 hrs for 20 wt.% SiC when compared with 14 hrs for unreinforced alloy for the same degree of deformation. Also, with 30% deformation the peak hardness values of the unreinforced and composite alloys are found to shift to shorter times, Table 1.

As a conclusion, it can be suggested that accelerated effect due to the addition of reinforcement is less than that due to the effect of deformation, Table 1. This means that the peak hardness of deformed materials is found to start at shorter times than that of the undeformed ones for both 6061 Al-alloy and composite materials.

.Thermo-mechanical treatment (TMT):-

The effects of cold deformation after solutionizing and natural aging for 24 hrs, on the age-hardening curves followed by final aging at 170°C for unreinforced and reinforced with 10 wt.% SiCp are shown in Figs 9-11. These curves show that a rapid response to artificial aging in both composite and Al-alloy followed by smooth increase in hardness with increasing aging time up to maximum after which it decreases with further increase in aging time.

The results show that the peak hardness values for unreinforced alloy are higher than the reinforced alloys. It is obvious that the total level of hardening attained via aging is very much reduced in the composites in comparison with the unreinforced Al-6061 matrix, Figs. 9-11

On the other hand, the precipitation in TMT was accelerated in both composites and aluminium alloy and hence, resulted in shorter time to attain the peak hardness. It can also be seen that the greater the amount of deformation (10-30 %), the more rapid is the acceleration of the aging process for both reinforced and unreinforced alloys, Table 1.

The preaging at room temperature has some effect on the reduction of hardness values of the composite alloys compared with the 6061 Al-alloy.

DISCUSSION:-

The 6061 Al Mg Si alloy is one of the most common aluminium based alloy as metallic matrix in discontinuously reinforced aluminium based metal matrix composites[1,15]. In order to provide insight into the aging results obtained in the present study, some background information on the precipitation sequence of 6061 Al-alloy is provided here. The precipitation sequence of reinforced 6061 Al-alloy have been shown to be identical to those of unreinforced 6061 Al-alloy[14, 16, 17] namely:-

$$\text{Supersaturated solid solution} \rightarrow \text{G.PZones} \rightarrow \beta' \rightarrow \beta\ (Mg_2Si)$$

The β' phase is needle-shaped, metastable and semi-coherent, while β phase is platelet-shaped, stable and incoherent.

In general, studies indicate that GP zones precipitates are relatively stable and are the only precipitate structures present even after any aging times at room temperature.

Temperature at 170°C or above are required to precipitate β'. Recent work on SiC particle reinforced 6061 has indicated that accelerated aging is associated with the development of β' precipitates and that the growth of GP zones is unaffected by SiC particles.

Therefore, for various heat treatment used, the aging at room temperature indicate that GP zones precipitates are formed while at 170°C aging should involve the β' precipitate.

In particular, the composite strengthening at room temperature was significantly lower than that observed in the three other heat treatment conditions.

Also, the results of the aging studies revealed an increase; in aging kinetics of the matrix with an increase in wt% of SiC particulates. The accelerated aging behaviour observed in the present study can be attributed to the enhanced heterogeneous nucleation of the strengthening phases in the metallic matrix during the aging step of the conventional T_6 heat treatment. The enhanced heterogeneous nucleation of the strengthening phases in the metallic matrix can primarily be attributed to:

a) The presence of SiC particulates,

b) Formation of defect-rich interfacial region in the near vicinity of SiC particulates, and;

c) Modification of microstructure features in the metallic matrix due to the presence of SiC particulates.

The ability of the SiC particulates to act as heterogeneous nucleation sites is consistent with the research findings of other investigators showing convincingly the precipitation of secondary phases at the SiC interface in some Al-alloys[18,19]. The heterogeneous nucleation capability of interfacial region formed in the near vicinity of SiC particulates can be attributed, in part, to the high dislocation density present in the composite matrix arising due to the mismatch between the coefficients of thermal expansion of the metal matrix and the ceramic reinforcement[20,21]. This increase in dislocation density promotes the dislocation assisted diffusion of the alloying elements from the adjacent dislocation lean areas of the matrix resulting into the solute enrichment in the interfacial region thus making the compositional requirement for the precipitation more favorable. This heterogeneous nucleation volume or plastic zone[22] formed around SiC particulates as a result of the variation in coefficient of thermal expansion between SiC particulates and the metallic matrix plays an instrumental role in heterogeneous nucleation of strengthening phases. The heterogeneous nucleation capability of the plastic zone can primarily be attributed to the presence of high dislocation density and relatively finer subgrain size[11]. In addition, it has also been suggested that the distortion field surrounding edge dislocations reduce the energy barrier for nucleation of the β' phase, efficiently increasing aging kinetics[11,23].

Finally, the accelerated aging kinetics observed in the present study can also be attributed to an increase in subgrain and grain boundary area in the metallic matrix. The decrease in subgrain and grain size of metallic matrix as a result of the presence of ceramic reinforcement has been previously established by various investigators[11,16].

An increase in grain boundary area in the matrix will assist in increasing the frequency of nucleation of strengthening phases as a result of the reduced activation barrier for the heterogeneous nucleation[11].

On the other hand, the results show that the peak aged hardness of the composite alloys is comparable with or even less than, that of the unreinforced Al-alloy. The smaller degree of hardening observed in the composite alloys can be attributed to the smaller amounts of precipitate forming elements in the composite matrix as compared with monolithic alloy. Besides, a possible reaction has been proposed by a number of authors[24,25] explain the presence of an $MgAl_2O_4$ spinel at the surface of SiC reinforcements in aluminium matrix composite. This is:

$$2\,SiO_2 + 2\,Al + Mg \rightarrow Mg\,Al_2O_4 + 2\,Si \qquad (1)$$

Where silica originates from the silica rich surface layer which is often present naturally on silicon carbide reinforcements. This observation of the spinel phase has been clearly linked with depletion of magnesium from an MMC matrix by some investigators[26,27]. This spinel formation has several potential disadvantages[28].

a) It removes Mg from the matrix, and hence reduces the age hardening capability of the matrix.

b) It increases the viscosity of the melt.

c) It may decrease the particle/matrix interfacial strength.

The results show that the existance of a reaction between the matrix and the SiC reinforcements has had a significant effect on magnesium depletion and hence in the Mg/Si ratio in the matrix of the MMC. In this work, the evidence of magnesium depletion comes indirectly from measuring the hardness developed during aging. Generally, the amount of hardening produced will depend primarily on the amounts of magnesium and silicon available for Mg_2Si formation. In the present work, it is the magnesium contents, which determine the amount of Mg_2Si formed because silicon is present in excess. Low hardness corresponded to low magnesium content, i.e., very little Mg_2Si precipitation.

At the same time, the results show that cold deformation accelerates aging that caused by the reinforcement addition. The drop in hardness at the beginning of aging curves can be attributed to the effect of elimination of residual stresses and relaxation of softening distortion which is more predominant in the initial stage of aging[11]. At the same time, the deformation after quenching and before aging altered aging precipitation sequence significantly. Therefore, the enhanced dislocation density in the 6061 Al alloy and its composite after cold deformation accelerated β' formation. The more degree of deformation, the higher dislocation densities and hence, the faster was the precipitation.

Generally, it can be observed that an increase in the dislocation density in the matrix because of cold rolling resulted in accelerated aging.

CONCLUSIONS:-

1- The presence of SiC particles decreases the grain size of the matrix. The higher the percentage of SiC particles, the smaller is the grain size.

2- The precipitation behaviour of the composite alloys is greatly different from that of unreinforced 6061 Al-alloy, despite the fact that the two alloys have similar chemical compositions.

3- The hardening during natural aging of both 6061 and its composite is due to the formation of GP zones while aging carried out at 170°C basically causes the formation of the needle shaped phase and the concomitant partial precipitation of the rod shaped phase β'.

4- The peak hardness for composite alloys is comparable or less with that for 6061 Al-alloy. The depletion of magnesium in 6061 Al matrix composite results in a reduction of the hardening produced during aging. This would reduce the amount of magnesium available in the composite matrix for β' formation during artificial aging at 170°C.

5- The presence of SiC particulates in Al-matrix accelerates the aging kinetics by comparison with the unreinforced matrix. Also, cold deformation accelerates aging similar to that caused by the reinforcement addition. It is possibly due to the fact that cold deformation produced a larger number of dislocations, which are evenly, distributed throughout the matrix.

6- The precipitation in TMT was accelerated in both composites and 6061 Al-alloy and hence, resulted in shorter time to attain the peak hardness, the greater the amount of deformation the more rapid is the acceleration of the aging process for both reinforced and unreinforced alloys.

REFERENCES:

I- I. A. Ibrahim, F. A. Mohamed, and F. J. Lavernia; J. Mat. Sci., 26 (1991) 1137

2- M.K. Surappa, J. Mat. Proc. Tech., 63 (1997) 325

3- T Christman, and S. Suresh, Acta Met., 36 (1988) 1691

4- I. Dutta, and D.L. Bourell, Mat. Sci. Eng., Al12 (1989) 67

5- S.J. Harris, H.W. Gai, and P.C. Weatherbern, (paper presented at the 93 Int. Conf. In Advances in Composite Materails, T.M. S., (1993)

6- T.G. Nieh, and R.F. Karlark, Scripta Met., 18 (1984) 25

7- M. Vogelsang, R.J. Arsenault, and R. Fisher, Met. Trans.A., 17 (1986) 379

S- S. Ikeno, K. Kawashima, K. Matsuda, H. Anada, and S. Tada, J. Jpn. Inst. Light Met., 40(1990) 501

9- K. Kawashima, J. Jpn. Inst. Light Met., 41 (1991) 725

10- M. Tayer, K.E. Lvloy, and D.J. Lioyd, Acta Met., 39 (1991) 73

11- R.J. Arsenault, L. Wang, and C.R, Ferg, Acta Met., 39 (1991) 47

12-A.N. Abd El-Azim, and S.F. Moustafa, 6'h Yugoslav Intern. Symposium on Aluminium, Ljubljana, Yugoslavia, May 1990

13- D.J. Lloyd, H. Lagace, A. Mcleod, and P.L. Morris, Mater.Sci.Eng.,A107 (1989) 73

14- M.J. Hadian, Y.W.Mai, and J.C.Healy, J.Mat.Sci., 28 (1993) 3665

15- A.L. Geiger, and J.A. Wolker, J. Met., 43 (1991) 8

16- M. Gupta, T.S. Srivatsan, F.A. Mohamed, and E.J. Lavernia, J. Mat. Sci., 28 (1993) 2243

17- Y. Song, and T.N. Baker, Mat. Sci. And Tech., 10(1994)406

18- M. Gupta, and M.K. Surappa, Mat. Research Bulletin, 30(1995) 1023

19- D.J. Lloyd, Int. Mater. Rev., 39(1994)1

20- W.S. Miller, and F.J. Humphreys, Scripta Met., 25 (1991) 33

21- R.J. Arsenault, Scripta Met., 25 (1991) 2617

22- Y. Wo, and E.J. Lavernia, Scripta Met., 27 (1992) 173

23- I. Dutta, and S.L. Bourell, Acta Met., 38 (1990)2041

24- H. Ribes, M. Suery, G. L'esperance, and J.G. Legoux, Met. Trans. A, 21 (1990) 2489

25- D.J. Lloyd, H.P. Lagace, and A.D. Mcleod, in controlled interphases in composite materails, (ed. H. Ishida), New York, NY, Elsevier, (1990) 359

26- B.R. Henriksen, Composites, 21(1990) 333

27- R. Warren, and C.H.Li, in " Proceedings of the 3rd Inter. Conf On Composite Interfaces" Ohio, May 1990

28- L. Jim, and D.J. Lloyd, 2"d International conference on cast metal matrix composites, Dusceloose, Alobawa, (1996), 282

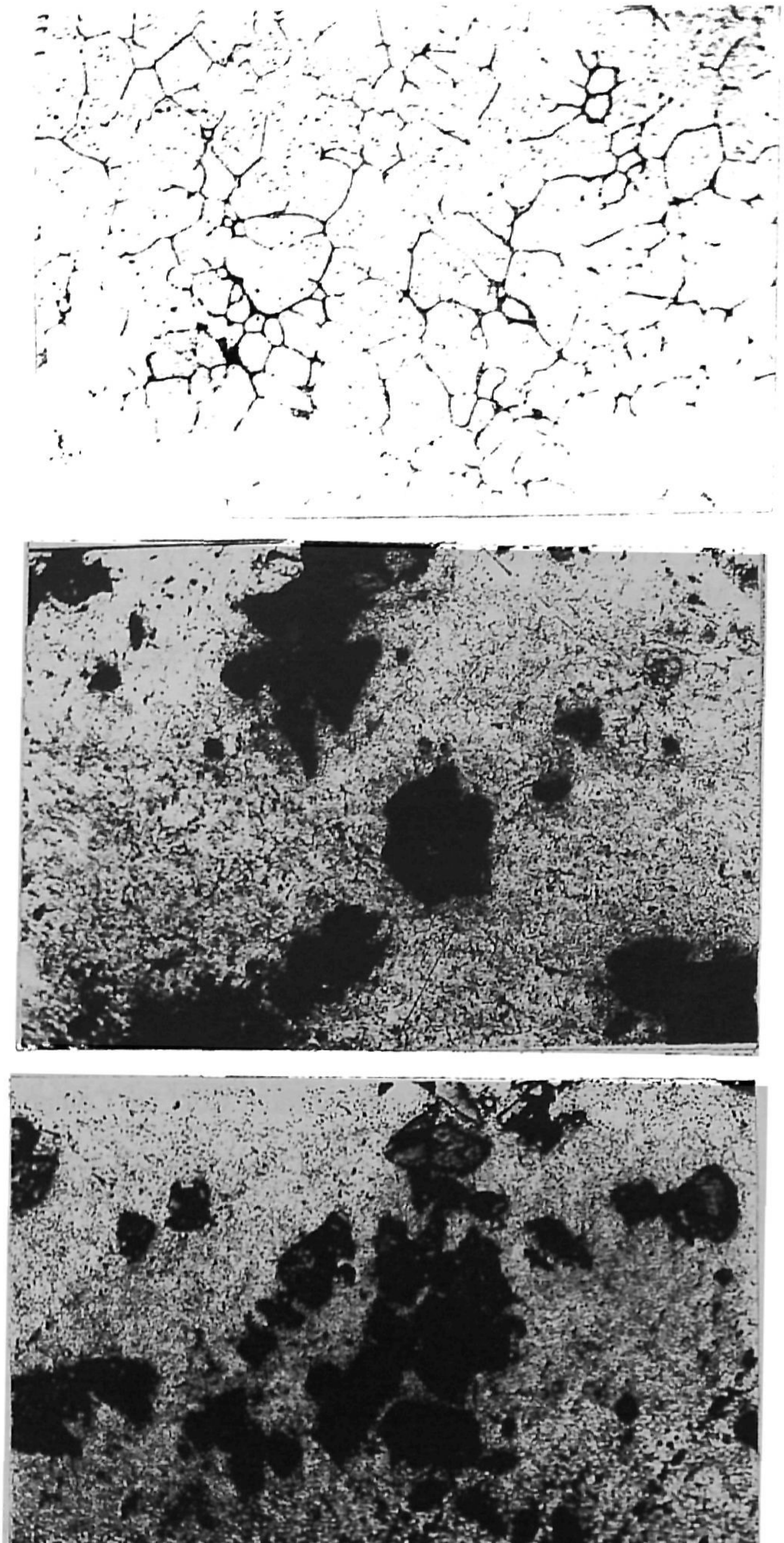

Fig. 1- Microstructure of the unreinforced and reinforced 6061 Al-alloy in cast condition.

a) 0 % SiC b) 5 % SiC c) 10 % SiC

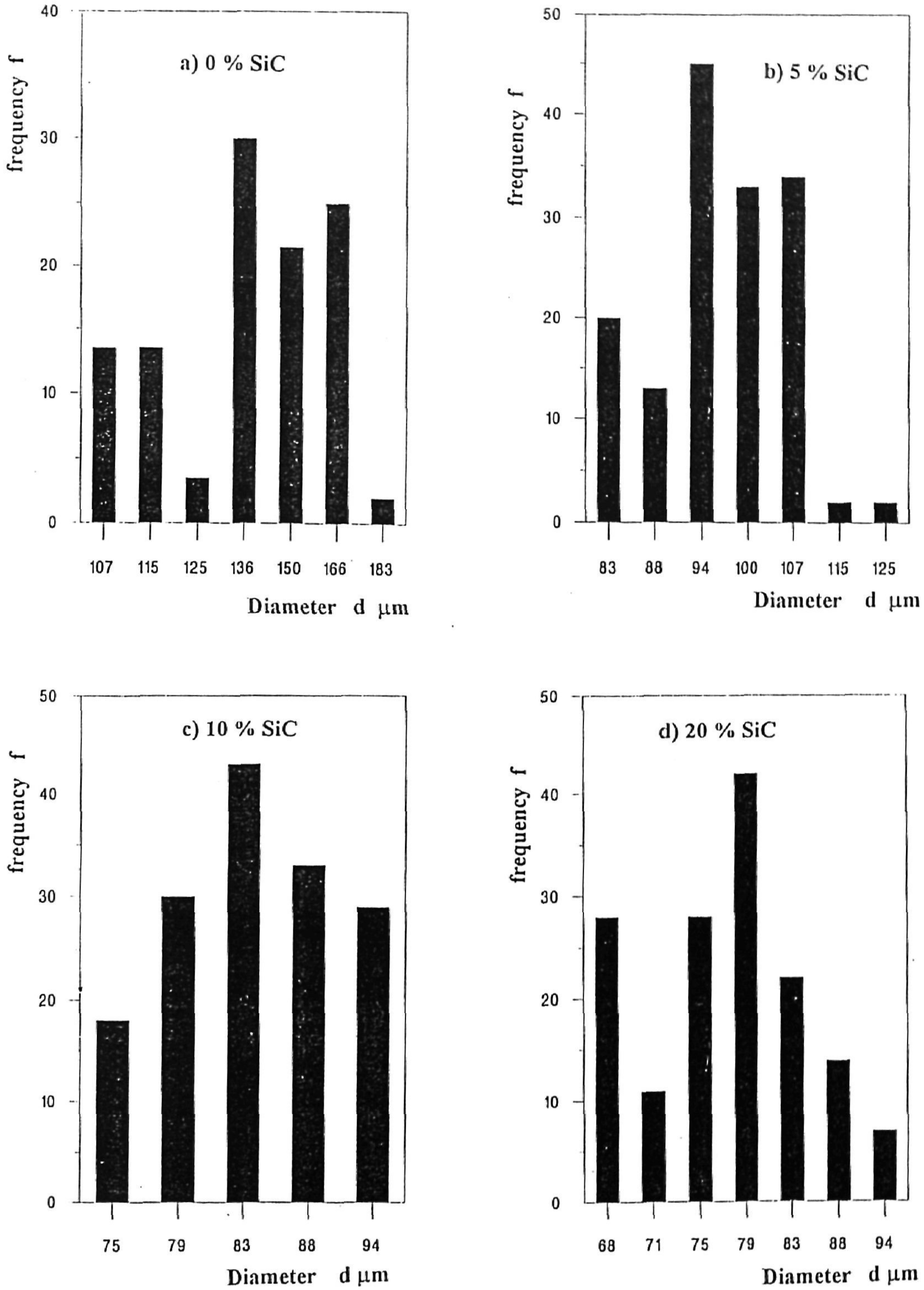

Fig. 2- Grain size distribution of both unreinforced and reinforced 6061 Al-alloy

Table (I)- Summary of Aging Response for composite and 6061 alloys.

System Studied	Reduction %	Aging Temp. (°c)	Time Taken For Peak HV (hr)	Peak Hardness (HV)	Remarks	
0 % Sic	—	25	128	68	Natural	Aging Hardening
5 % Sic	—	25	112	64		
10 % Sic	—	25	88	65		
20 % Sic	—	25	76	65		
0 % Sic	—	170	19	115	Artificial	
5 % Sic	—	170	17	110		
10 % Sic	—	170	14	114		
20 % Sic	—	170	12	114		
0 % Sic	10	25	124	63	Natural	Effect of degree of deformation on aging
5 % Sic	10	25	104	56		
10 % Sic	10	25	84	62		
20 % Sic	10	25	72	57		
0 % Sic	10	170	14	101	Artificial	
5 % Sic	10	170	12	92		
10 % Sic	10	170	11	92		
20 % Sic	10	170	10	90		
0 % Sic	30	25	112	75	Natural	
5 % Sic	30	25	96	81		
10 % Sic	30	25	76	84		
20 % Sic	30	25	68	88		
0 % Sic	30	170	11	106	Artificial	
5 % Sic	30	170	9	103		
10 % Sic	30	170	8	105		
20 % Sic	30	170	7	100		
0 % Sic	10	170	12	114	Pre-aging At room Temp. For 24 hrs and then cold deformation	Termomechanical Treatment (TMT)
10 %Sic	10	170	11	93		
0 % Sic	20	170	11	114		
10 %Sic	20	170	9	101		
0 % Sic	30	170	7	119		
10 %Sic	30	170	5	111		

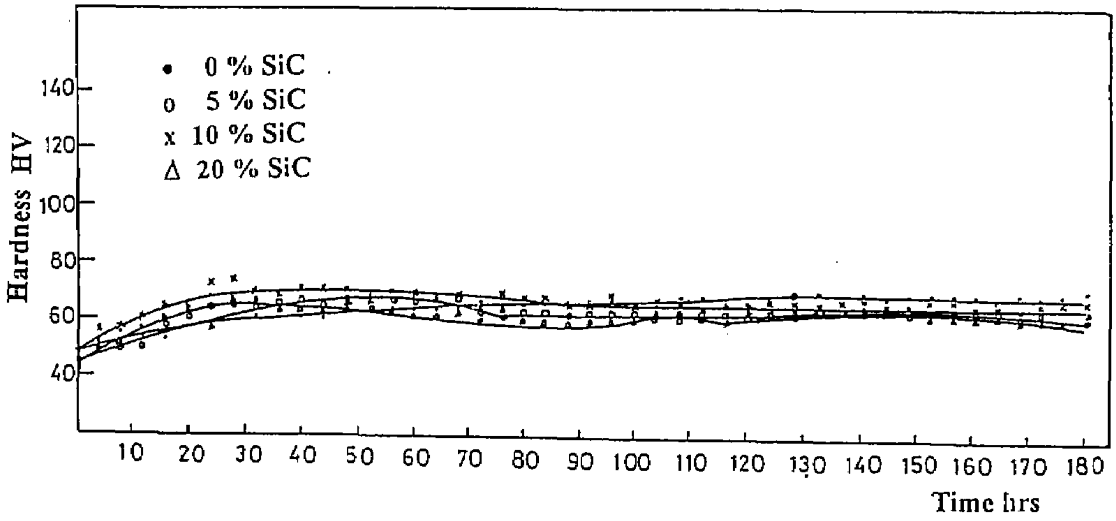

Fig. 3- Variation of hadness with time during room temperature aging

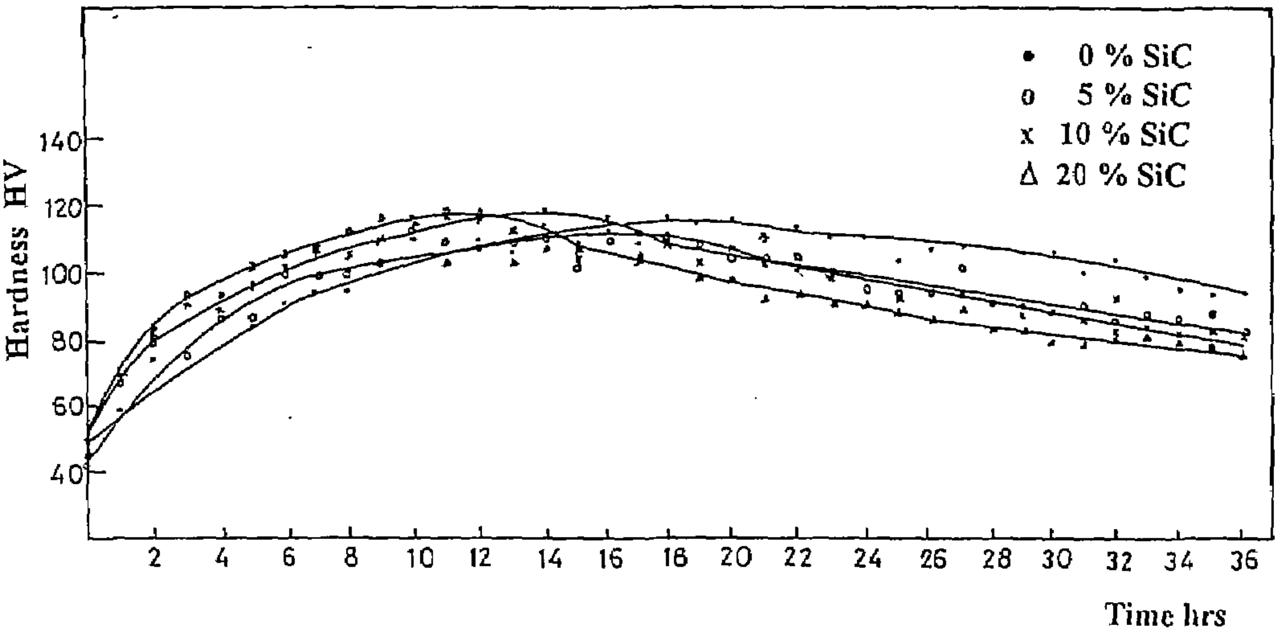

Fig. 4- Variation of hardness with time aging at (170°C)

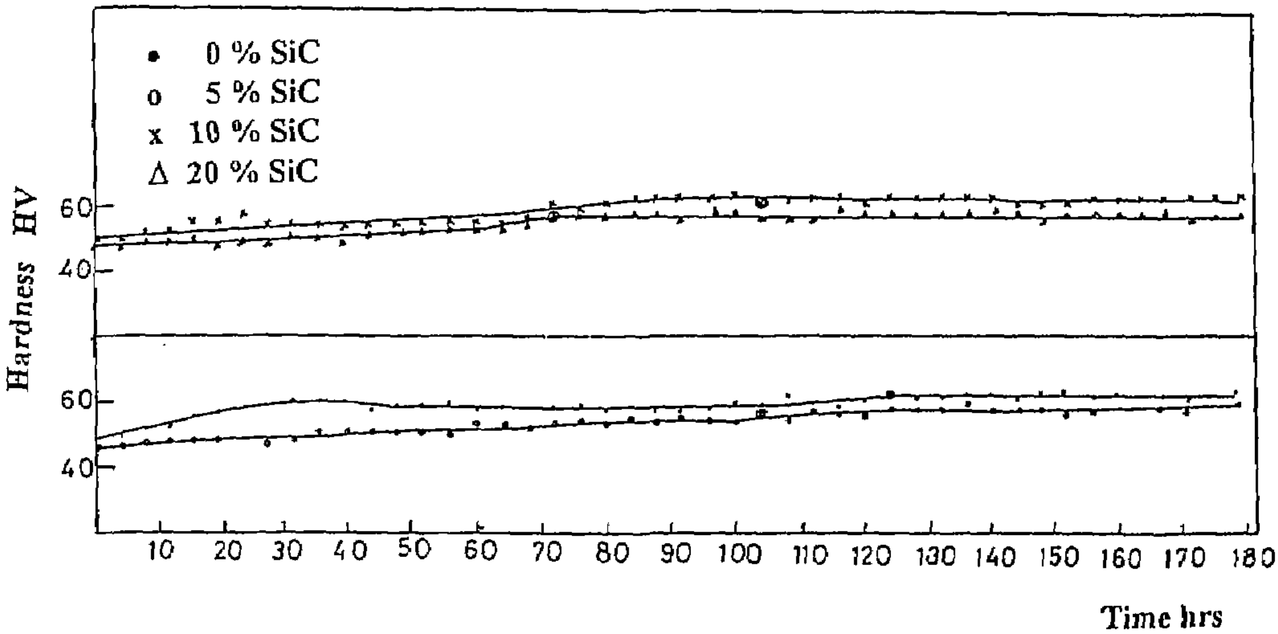

Fig. 5- Variation of hardness with time during room temperature aging after 10 % deformation

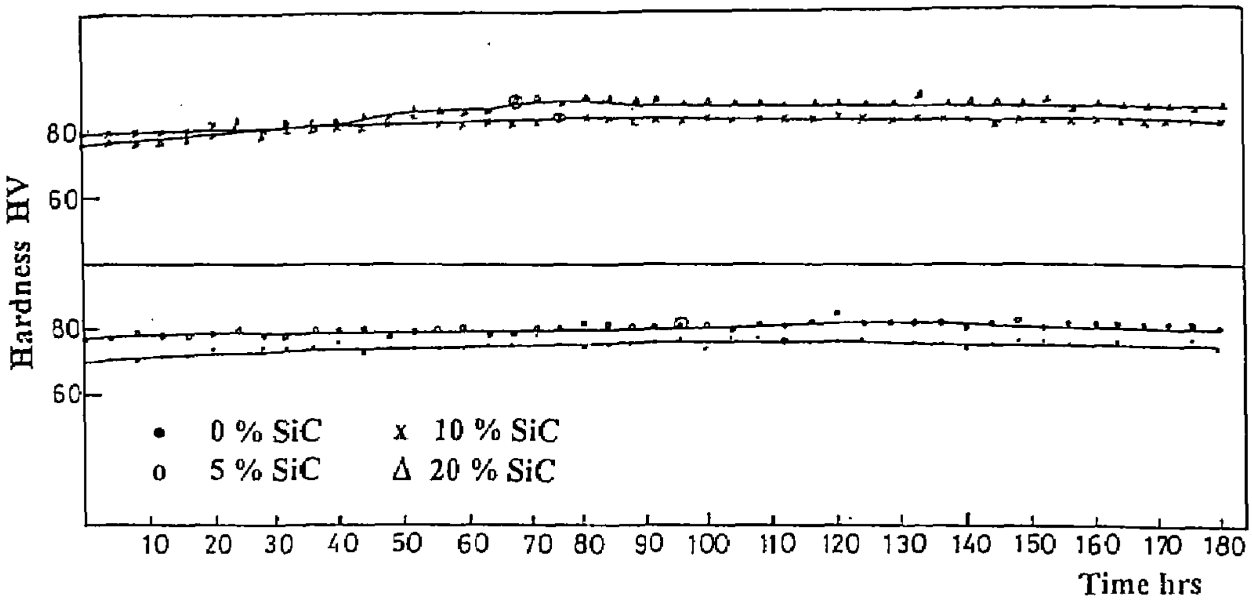

Fig. 6- Variation of hardness with time during room temperature aging after 30 % deformation

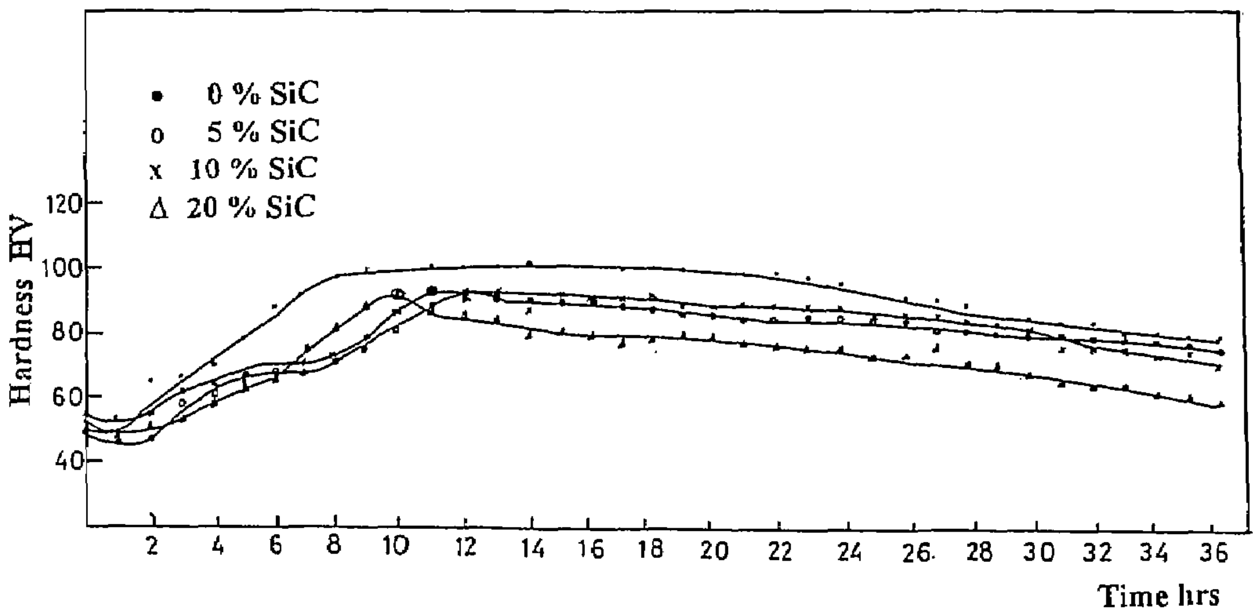

Fig. 7- Variation of hardness with time during aging at (170°C) after 10 % deformation

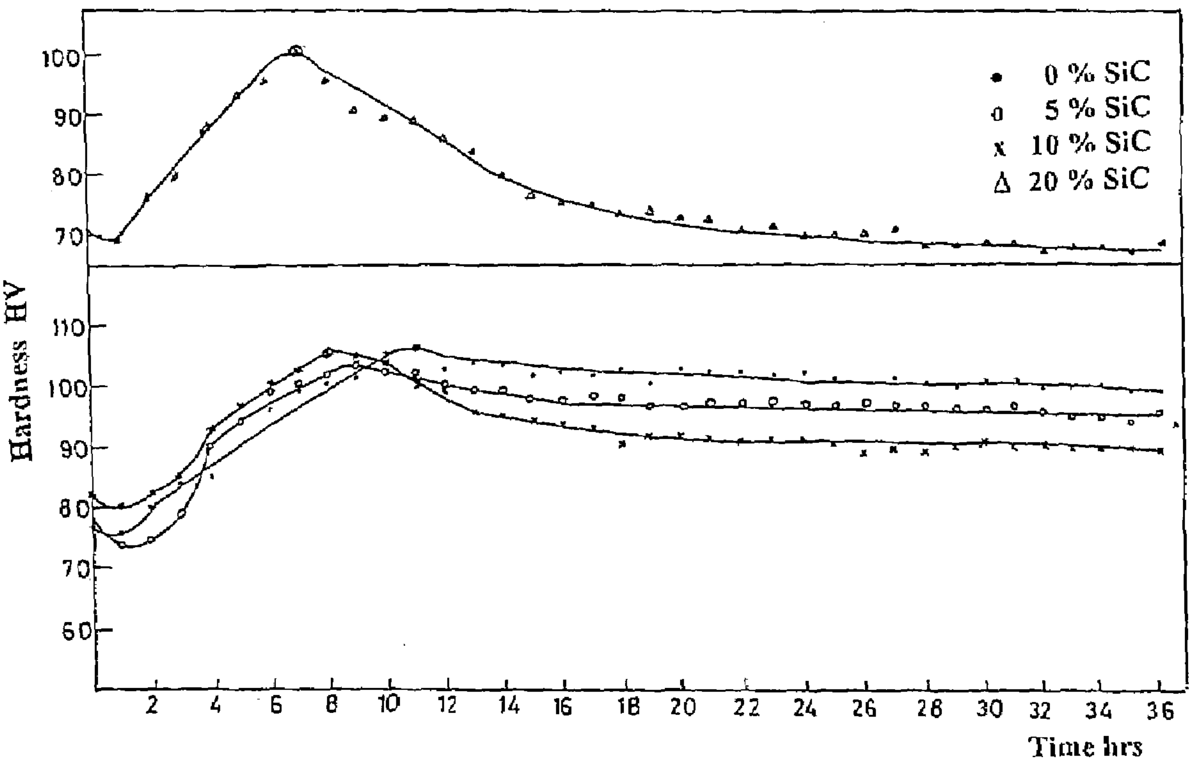

Fig. 8- Variation of hardness with time during aging at (170°C)

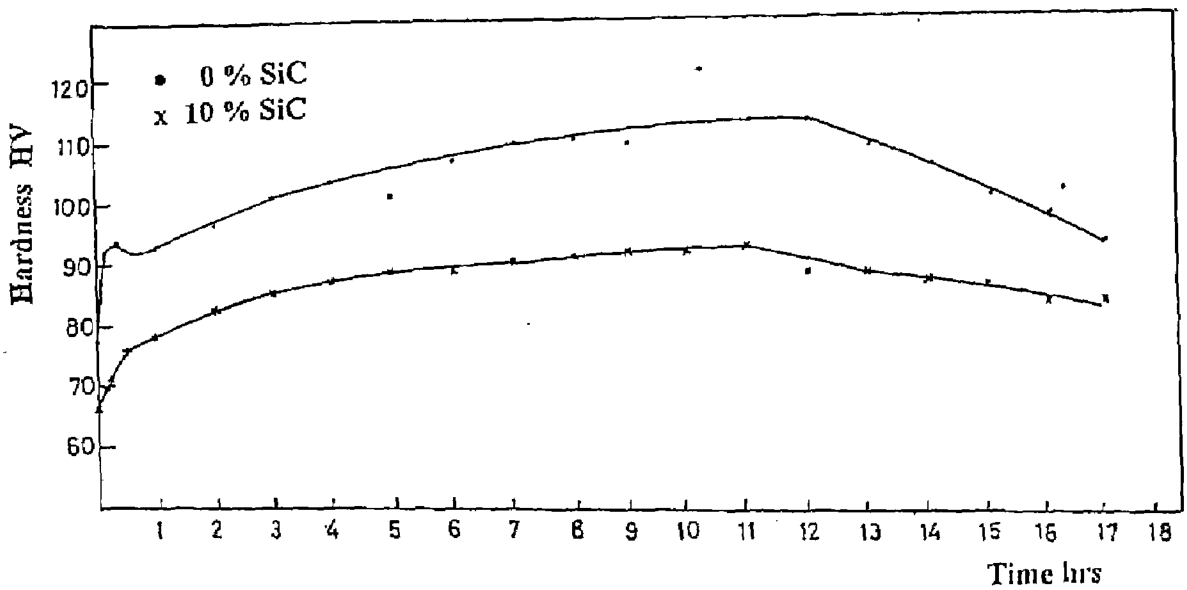

Fig. 9- Variation of hardness with time during aging at (170°C) after TMT process (10 % def.)

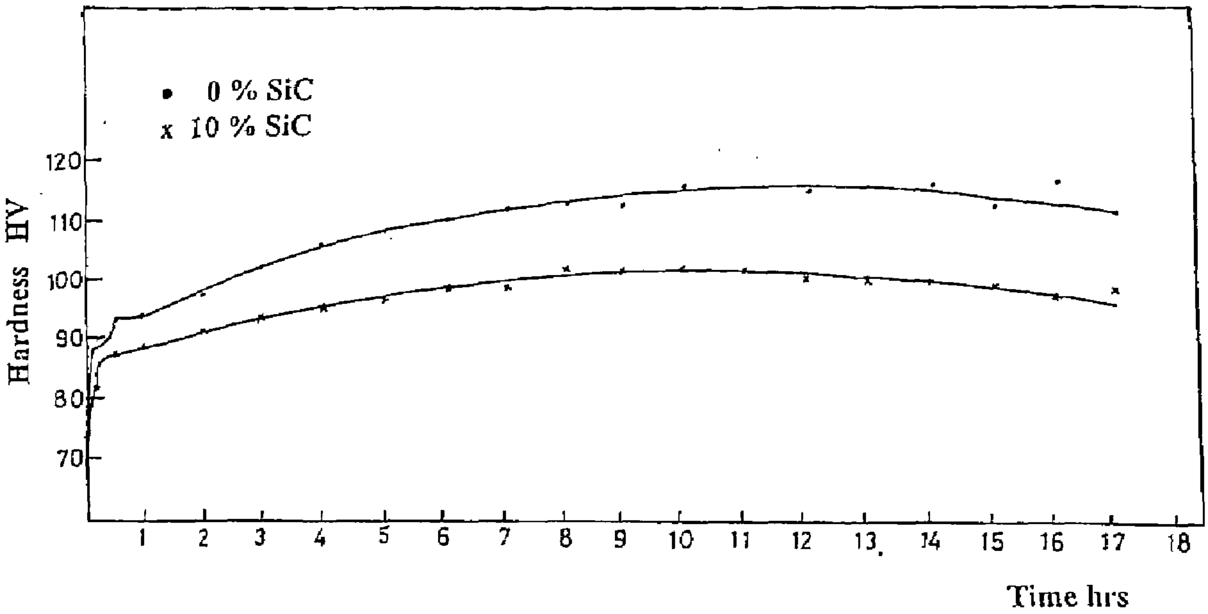

Fig. 10- Variation of hadness with time during aging at (170°C) after TMT process (20 % def.)

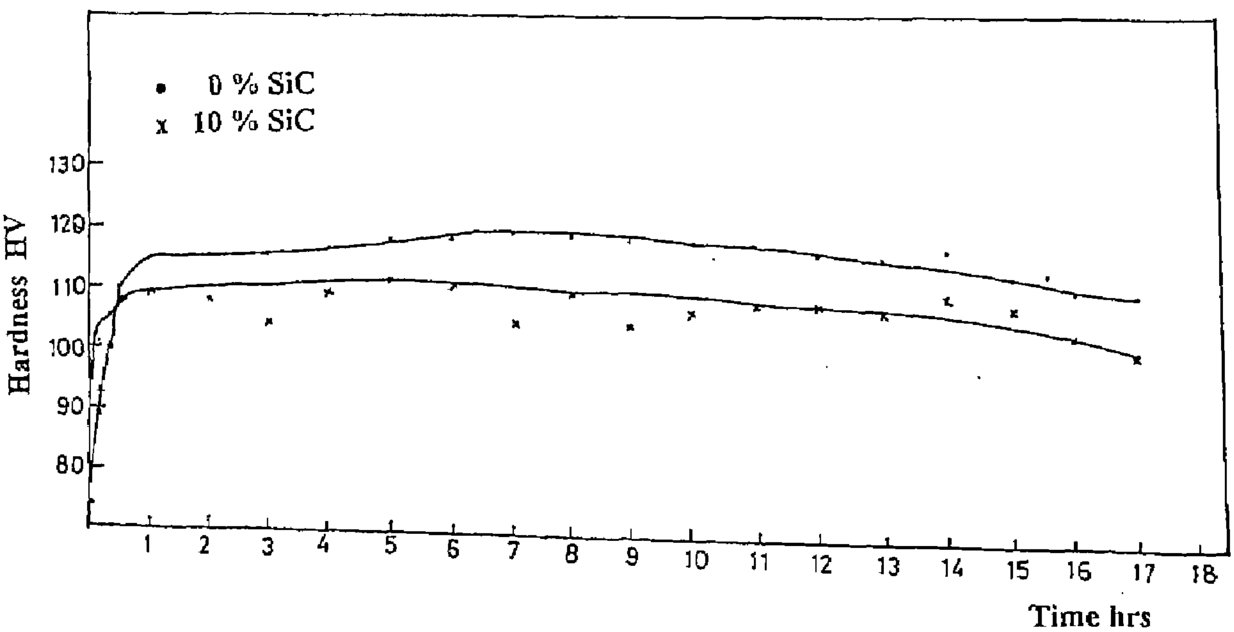

Fig. 11- Variation of hardness with time during aging at (170°C) after TMT process (30 % def.)