Some Observations on Hardness Measurements of Particulate-Reinforced 6061 Aluminum Metal Matrix Composites

T. Das, S. Bandyopadhyay, and S. Blairs

Hardness measurements on a series of particulate-reinforced metal matrix composites in a solution treated and T-6 condition were carried out using a Vickers microhardness tester at 25-, 50-, 100-, 200-, 300-, and 500-g indenter loads and a Vickers macrohardness tester at an indenter load of 5 kg. It appears that the presence of the particles makes a contribution to the hardness measurement, the degree of which depends on the size and distribution of the particles, and also the indentation load. Although some trends are observed, there is no predictable effect of the material and test parameter on the hardness values.

1. Introduction

PARTICULATE-REINFORCED metal matrix composites (PMMC) are receiving increasing attention in the literature as potentially important engineering and structural materials,[1] because of their light weight and improved strength and stiffness over the light alloy matrix. The majority of particulate-reinforced metal matrix composite materials has been developed using heat treatable aluminum alloys. Several investigators have used hardness or microhardness (Brinell, Vickers, Knoop, Rockwell B)[2-8] as a parameter to correlate strength with degree of age hardening of the matrix in these metal matrix composites. Such studies have relevance to the fact that the matrix strength has been considered a primary parameter influencing the strength of composites.[9] However, it should be recognized that, in composites, particularly with small size reinforcement, the hardness observed may not necessarily be the true hardness of the matrix, as there may be a hardness contribution by the particles. For this reason, the hardness of the composite may be greater than that of the matrix. This could mistakenly imply that, in the presence of particles, the matrix age hardens to a greater hardness than without particles.[7,8] Consideration should thus be given to the microscopic deformation processes involved with the application of a load and subsequent interpretation of hardness values in particulate-reinforced composite materials. This aspect appears to have been neglected by most of the workers.[2-8]

As part of a broad research program on particulate-reinforced metal matrix composite materials in the authors' School, attention was given to the measurement of hardness in a series of particulate-reinforced metal matrix composite materials based on a 6061 aluminum matrix. This article presents results of those studies using fine SiC and coarse Al_2O_3 particulate reinforcement.

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2. Materials and Methods

2.1 *Materials*

The materials used in the study were 19-mm diameter rod, extruded from billets containing (1) 10 and 20% volume fraction (V_f) fine SiC/6061 composites manufactured by a powder metallurgy route (California Consolidated Technology), (2) 10 and 20% volume fraction coarse Al_2O_3/6061 composites produced by a liquid metallurgy route (Duralcan), and (3) 20% volume fraction microspherical Al_2O_3/6061 composites (Comral-85) produced by Comalco Technology. The first four materials were procured as larger diameter billets by Comalco, who then extruded them to 19-mm diameter rod.

For hardness measurements, specimens approximately 15 mm in length were cut from the rods and polished on various grades of SiC paper (up to 1200 grit). Final polishing was carried out using 3-μm diamond abrasive, 1-μm diamond abrasive cloth, and then finally a silica suspension on the polishing pad. Hardness measurements were carried out on specimens in both the solution treated as well as T-6 condition (530 °C for 1.5 h,

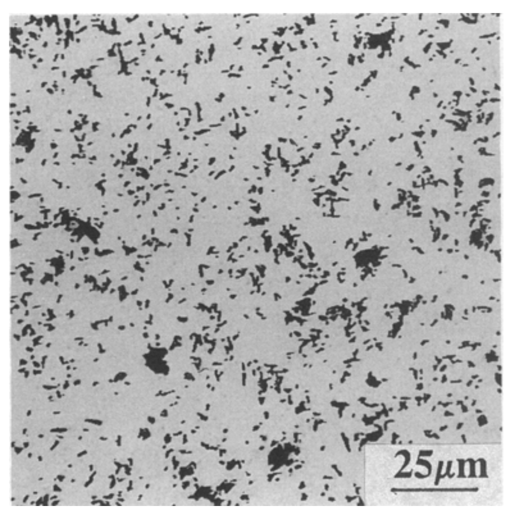

Fig. 1 Optical micrograph of 6061 + 10% SiC.

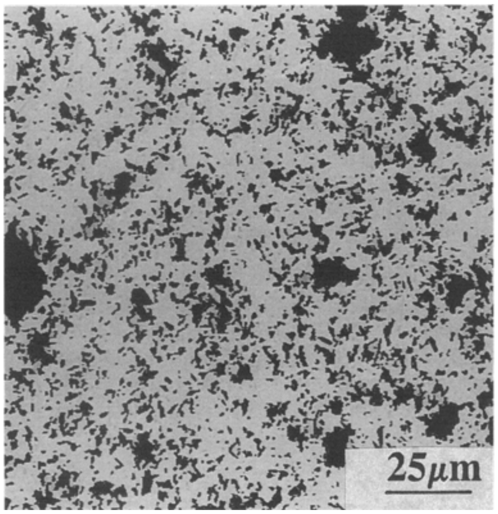

Fig. 2 Optical micrograph of 6061 + 20% SiC.

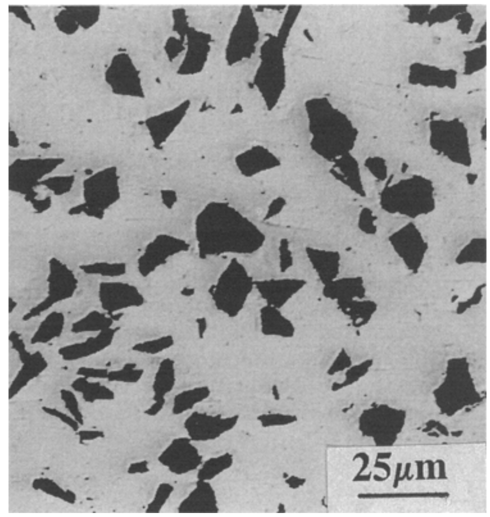

Fig. 4 Optical micrograph of 6061 + 20% Al_2O_3.

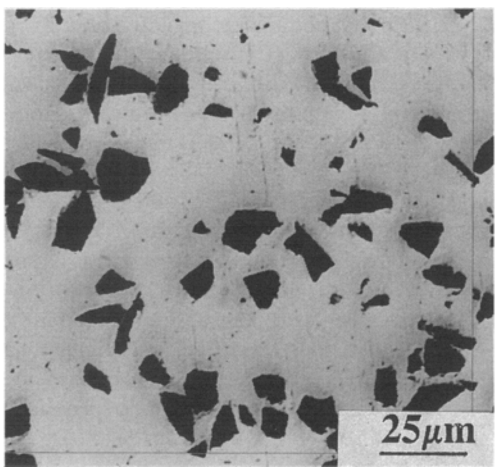

Fig. 3 Optical micrograph of 6061 + 10% Al_2O_3.

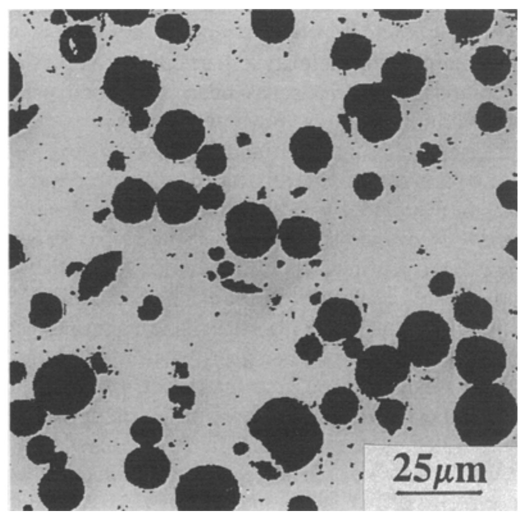

Fig. 5 Optical micrograph of 6061 + 20% Al_2O_3 (microspherical) (Comral-85).

cold water quenched, 20 h aging at room temperature, followed by thermal aging at 175 °C for 8 h). In addition to the five metal matrix composites, the hardness of an unreinforced Comalco 6061 aluminium 19-mm diameter rod was examined in the solution treated and T-6 conditions for comparison.

2.2 *Particle Size Analysis*

Particle size analysis of the materials was carried out using a Quantimet-570 Image Processing and Analysis System. The area under observation was 450 by 450 pixel, (1 pixel = 0.394 μm). The Quantimet-570 Image Analysis System accepts standard television signals from a television camera mounted on an optical microscope and displays processed images on a monitor.

2.3 *Hardness Measurements*

Hardness measurements were carried out using (1) a Durimet Vickers microhardness tester using indenter loads of 25, 50, 100, 200, 300, and 500 g and (2) a conventional Vickers (macro) hardness tester at a load of 5 kg. An average of five values was taken for each specimen at each load.

3. Results and Discussion

Typical microscopic distributions of particles for the five metal matrix composites are given in Fig. 1 to 5, and the analysis of the particle size distribution corresponding to these figures is given in Tables 1 and 2. Some overlapping zones are apparent in Fig. 1 (10% SiC) and in Fig. 2 in particular (20%

SiC). Table 1 provides a mean equivalent diameter of 2.38 μm for the 10% SiC/6061 and 2.37 μm for 20% SiC/6061 particulate-reinforced metal matrix composite. If particles 11 μm in size and above are ignored in the latter material, assuming these sizes are due to overlapping, one obtains a mean equivalent diameter of 2.2 μm.

No overlapping is observed in the Al_2O_3 composites, rather there are a number of fine particles present (Table 2). For these two materials, if all particle sizes (as in Table 2) are considered, then one obtains an average equivalent diameters of 8.46 and 5.29 μm, respectively, for the 10 and 20% Al_2O_3 materials. If the two lowest size fractions, *i.e.*, below 3 μm are ignored, then a more even average size results (10.76 and 11.7 μm). In a similar way, the 20% microsphere Al_2O_3 particulate-reinforced metal matrix composite (Comral-85) provides a mean particle size of 12.9 μm (rather than 4.6 μm if all the fractions are taken into account). Comalco observed an average particle size of 20 μm for this material using the free particles. However, in a composite, one may not always see the diagonal sections of the reinforcement.

It is emphasized that this manipulation of data is done mainly to arrive at an average size of, presumably, the starting particles, but it is recognized that in the composites, the particle distributions are as given in Tables 1 and 2.

Results of the microhardness and macrohardness studies are given in Table 3. Several aspects of the microhardness measurements can be noted. First, there is some degree of load dependence of the hardness values in some materials, although the trend is not consistent. For example, in the 20% SiC/6061 material, hardness generally decreases with increasing indenter load, whereas for 10 and 20% Al_2O_3/6061 composites, the opposite is observed. In the case of 20% Al_2O_3 (microsphere)/6061, hardness is relatively constant up to 200 g load, then falls at higher indenter loads. 10% SiC/6061 and unreinforced 6061 alloy do not show any definite trend with indenter load, rather they display what is more likely a scatter in results.

Secondly, the 20% SiC particulate-reinforced metal matrix composite exhibits higher hardnesses than the rest of the materials, apparently more so in the as solution treated state, and to a lesser extent in the T-6 condition. Also, the macrohardness of the different materials is similar, both for solution treated material and for material in the T-6 condition, the general exception again being the 20% SiC-6061 materials in the solution treated

Table 1 Particle Size Distribution for 10 and 20% Volume Fraction Reinforced 6061 Alloys

Size distribution/ diameter, μm	10% SiC/6061	20% SiC/6061
D_p <1 μm	60	219
1 μm ≤ D_p < 2 μm	310	484
2 μm ≤ D_p < 3 μm	170	190
3 μm ≤ D_p < 4 μm	66	85
4 μm ≤ D_p < 5 μm	48	49
5 μm ≤ D_p < 6 μm	15	32
6 μm ≤ D_p < 7 μm	5	13
7 μm ≤ D_p < 8 μm	3	13
8 μm ≤ D_p < 9 μm	2	8
9 μm ≤ D_p < 10 μm	4	2
10 μm ≤ D_p < 11 μm	2	6
11 μm ≤ D_p < 12 μm	0	1
12 μm ≤ D_p < 13 μm	0	2
13 μm ≤ D_p < 14 μm	0	4
14 μm ≤ D_p < 15 μm	0	0
15 μm ≤ D_p < 16 μm	0	0
16 μm ≤ D_p < 17 μm	0	1
17 μm ≤ D_p < 18 μm	0	1
18 μm ≤ D_p < 19 μm	0	0
19 μm ≤ D_p < 20 μm	0	1
20 μm ≤ D_p < 21 μm	0	1
28 μm ≤ D_p < 29 μm	0	1

Note: From Fig. 1 and 2, respectively.

Table 2 Analysis of Particle Size Distribution of 10% Al_2O_3/6061, 20% Al_2O_3/6061, and 20% Al_2O_3 (Microspherical)/6061 (Comral-85)

Size distribution/ equivalent diameter (D_p)	10% Al_2O_3/6061	20% Al_2O_3/6061	20% Al_2O_3 (microspherical)/6061
D_p < 1 μm	3	37	36
1 μm ≤ D_p < 3 μm	14	52	92
3 μm ≤ D_p < 5 μm	7	11	9
5 μm ≤ D_p < 7 μm	10	5	6
7 μm ≤ D_p < 9 μm	5	10	5
9 μm ≤ D_p < 11 μm	6	3	5
11 μm ≤ D_p < 13 μm	4	5	2
13 μm ≤ D_p < 15 μm	8	7	3
15 μm ≤ D_p < 17 μm	2	6	4
17 μm ≤ D_p < 19 μm	5	3	1
19 μm ≤ D_p < 21 μm	1	3	1
21 μm ≤ D_p < 23 μm	1	2	3
23 μm ≤ D_p < 25 μm	...	1	2
25 μm ≤ D_p < 27 μm	...	...	2
27 μm ≤ D_p < 29 μm	...	...	2
29 μm ≤ D_p < 31 μm	...	...	1
31 μm ≤ D_p < 33 μm	...	...	...
33 μm ≤ D_p < 35 μm	1	...	...
35 μm ≤ D_p	...	...	1

Note: From Fig. 3, 4, and 5, respectively.

condition, which exhibited much greater hardness than the other materials in the solution treated condition.

It should be noted that the standard deviation of the microhardness values (Table 3) is low and consistent at higher indenter loads, *i.e.*, 200 to 500 g, for almost all the materials. Specific instances where the standard deviation of the hardness values is high include 10 and 20% SiC materials and the 10% Al_2O_3 composites at 25- to 100-g indenter loads. Macrohardness results, on the other hand, are very reproducible. Figures 6 to 9 present microhardness indentations for 10 and 20% SiC, 10% Al_2O_3, and 20% microspherical Al_2O_3 composites, where an indenter load of 50 g was used. It can be seen from these figures that, for the 10 and 20% SiC particulate-reinforced metal matrix composites, particularly for the latter, the indenter encounters a number of SiC particles. In the case of the Al_2O_3-containing materials, because of the large mean free path between particles, one is able to make indentations in the matrix, thus avoiding any direct contribution by the physical presence of the particles themselves, except when a higher indenting load is used.

The load dependence of Vickers microhardnesses is complex and is not clearly understood even in monolithic metals.[10] In the case of a particulate-reinforced metal matrix composite material, the presence of the second phase particle may introduce further degrees of complication. Although size, size distribution, and mean free path of particles play an influencing role on microhardness, the presence of particles below the indentation cannot be neglected, which can affect the results.

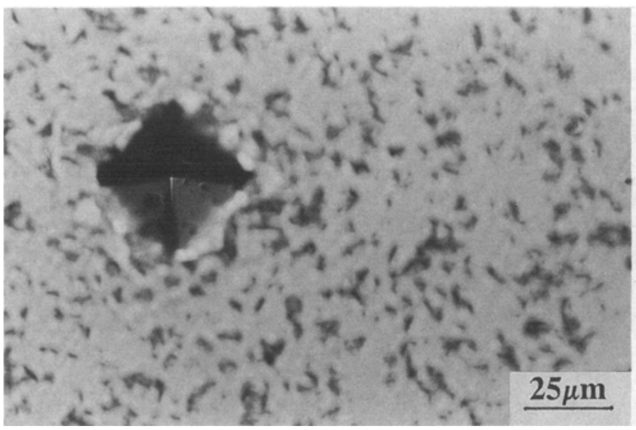

Fig. 6 Microhardness indentation for 10% SiC/6061 (load = 50 g).

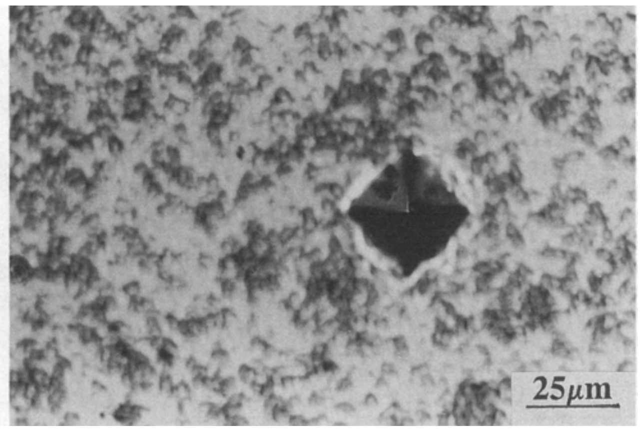

Fig. 7 Microhardness indentation for 20% SiC/6061 (load = 50 g).

Table 3 Vickers microhardness of metal matrix composites and unreinforced 6061 at various loads

Material/thermal treatment	Microhardness at various loads, HV 25 g	50 g	100 g	200 g	300 g	500 g	Macrohardness (HV), 5 kg/1000
10% SiC/6061:							
ST	70.8 (2.1)	69.5 (7.8)	67.2 (6.5)	66.6 (1.0)	68.3 (0.8)	67.3 (1.1)	66.6 (0.4)
T-6	111.0 (1.0)	114.2 (1.3)	113.0 (2.0)	103.8 (2.7)	106.2 (1.9)	108.6 (2.1)	110.6 (0.5)
20% SiC/6061:							
ST	88.2 (4.7)	81.5 (7.7)	83.8 (5.9)	79.4 (1.7)	77.9 (1.2)	77.8 (1.0)	76.8 (0.8)
T-6	119.0 (1.3)	121.8 (3.5)	124.4 (3.9)	117.2 (1.0)	115.2 (1.6)	117.0 (1.9)	116.2 (0.8)
10% Al_2O_3/6061:							
ST	54.5 (5.6)	56.7 (3.2)	60.1 (2.0)	64.9 (2.3)	63.7 (1.8)	64.6 (1.2)	62.4 (1.0)
T-6	110.4 (2.4)	105.0 (2.6)	105.8 (2.6)	100.9 (1.6)	103.4 (2.3)	105.8 (1.5)	106.4 (2.0)
20% Al_2O_3/6061:							
ST	57.1 (2.4)	57.0 (1.2)	58.1 (2.0)	64.9 (2.1)	65.8 (2.5)	68.0 (2.6)	60.7 (0.3)
T-6	111.8 (2.7)	106.8 (2.8)	105.6 (2.6)	108.4 (2.3)	111.4 (2.2)	113.4 (2.3)	110.6 (0.8)
20% Al_2O_3 (microspherical)/6061:							
ST	65.3 (4.4)	68.5 (1.3)	68.3 (1.6)	67.6 (2.7)	61.3 (0.8)	61.3 (1.1)	62.5 (0.4)
T-6	113.6 (1.9)	105.6 (3.1)	106.8 (3.5)	116.4 (3.5)	118.0 (1.1)	115.8 (1.5)	114.0 (0.9)
Unreinforced 6061:							
ST	65.2 (1.3)	62.9 (1.9)	60.2 (1.2)	62.8 (1.8)	61.3 (0.8)	61.3 (1.1)	60.6 (0.4)
T-6	118.4 (2.2)	115.0 (3.2)	115.4 (4.5)	108.0 (2.8)	112.2 (1.3)	106.4 (2.9)	116.6 (0.5)

Note: Numbers in parentheses denote standard deviation; ST = Solution treated.

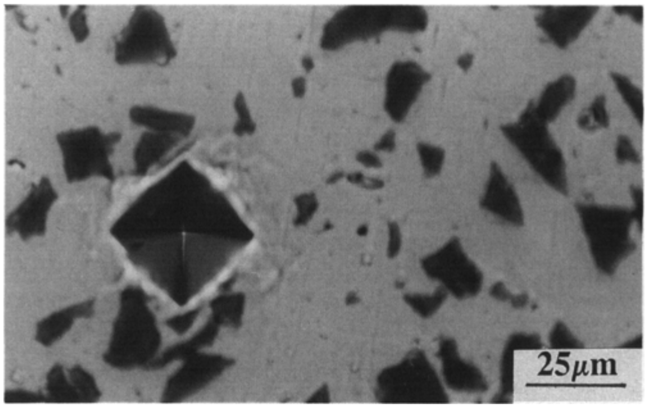

Fig. 8 Microhardness indentation for 20% Al_2O_3 (load = 50 g).

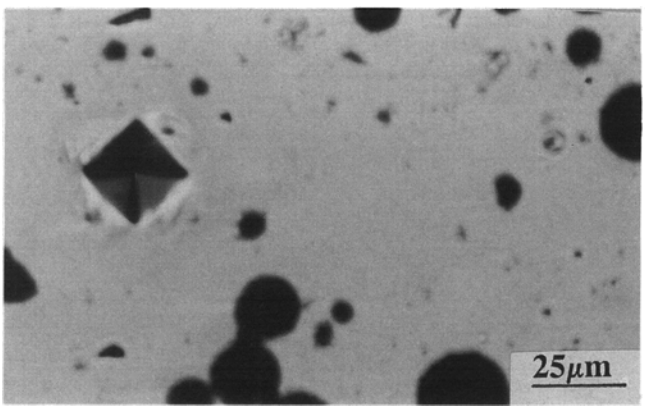

Fig. 9 Microhardness indentation for Comral-85 (load = 50 g).

In the case of fine particles, *i.e.*, 10 and 20% SiC-6061 materials, at all loads, the indenter will cover a number of particles. With the alumina-metal matrix composites, although at lower indenter loads it is possible to make indentations in the matrix without involving the particles, at higher loads, this is clearly not so.

Figures 10 and 11 show the variation in microhardness as a function of the ratio of indentation diameter (D) at various loads to the mean size of the particles (D_p) as obtained from Tables 1 and 2. In the case of the SiC/6061 materials, hardness is generally higher at lower loads and then starts to level off for indenter loads above ~100-g load. In the case of the Al_2O_3/6061 composites, however, no consistent trend is observed, but there is some tendency of increasing microhardness as the indenter load increases, except at very low loads. Presumably, this happens because, in these materials, indentations under higher loads involve reinforcement particles, whereas at lower loads a clean indentation can be obtained in the matrix. The effect of age hardening on hardness is quite clear in all the composites, but one cannot say with any certainty, from these results, if the

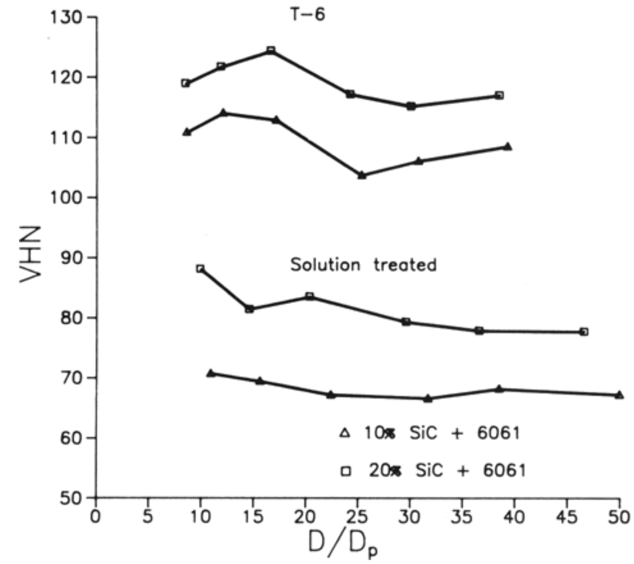

Fig. 10 Variation of microhardness as a function of the ratio of indentation diameter (D) to the mean size (D_p) of the particles for 10 and 20% SiC/6061 in T-6 and solution treated conditions.

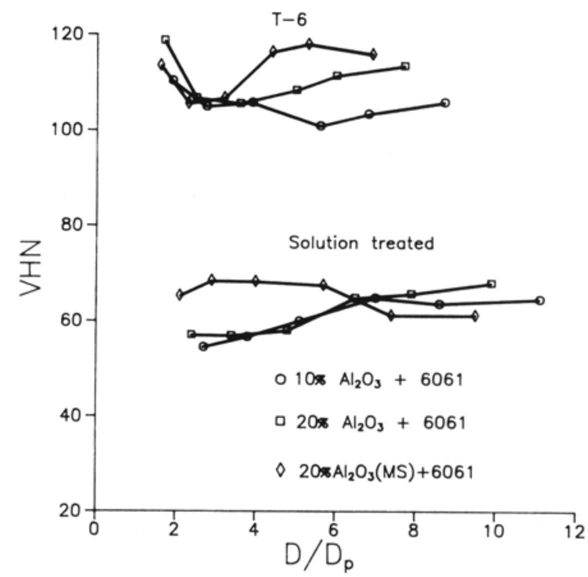

Fig. 11 Variation of microhardness as a function of the ratio of indentation diameter (D) to the mean size (D_p) of the particles for 10 and 20% Al_2O_3/6061 and Comral-85 in T-6 and solution treated conditions.

reinforcing phase enhances the kinetics of the thermal aging process.

4. Concluding Remarks

Interpretation of results of microhardness measurements for particulate-reinforced metal matrix composite materials should be treated with caution. The presence of the reinforcing particles, their size, size distribution, as well as mean free path, may have a contribution to hardness. Consequently, microhardness studies may not be used conclusively to study the influence of the particulate phase on the kinetics of the age hardening process.

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