

1 Whiskers of Al₂O₃ as reinforcement of a powder metallurgical 6061
2 aluminium matrix composite

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14
15 **Abstract**

16 An Al-Mg-Si alloy matrix composite reinforced with 10% volume of alumina whiskers
17 (Al₂O₃w) has been processed by powder metallurgy and investigated. The Al₂O₃w were
18 produced as single crystal c-axis alpha alumina fibres at pre-pilot scale via Vapour Liquid Solid
19 (VLS) deposition in a cold-wall air-tight furnace with alumina linings. As far as we know, this
20 is the first report of the utilization of whiskers of Al₂O₃ as reinforcing elements for Al alloys.
21 Tensile tests have been performed on the composite at room and high temperature. Results show
22 that the AA6061 alloy reinforced with the as-produced Al₂O₃ whiskers has remarkably high
23 mechanical properties at room temperature. This is attributed to the high quality of the Al₂O₃
24 single crystals and to the strong bonding attained between them and the 6061 alloy matrix.

25
26 *Keywords:* Composite materials; Microstructure; Mechanical properties; Aluminium, Alumina
27 whiskers; Powder metallurgy.

28

29 **1. Introduction**

30 Particle-, whisker- and fibre-reinforced metal matrix composites can sustain higher loadings
31 than the unreinforced equivalent matrixes. Whiskers are defect-free, very thin single crystal
32 fibres, which are among the most resistant materials known. Among ceramic whiskers, α -Al₂O₃
33 grown with c-axis orientation are good candidates for use as strengtheners in composites
34 because of their high elastic modulus, thermal and chemical stability, fracture strength, and
35 creep resistance even at high temperatures [1-3]. However, the use of Al₂O₃ whiskers has been
36 hindered up to now by its extremely high production cost. Accordingly, studies on metal matrix
37 composites reinforced with Al₂O₃ has been limited to particles, see for example ref. [6, 7], and
38 short fibres [8-11].

39 In this work, a novel method has been used for producing enough amount of c-axis α -Al₂O₃
40 whiskers, based on vapour-liquid-solid deposition (VLS) in Ar atmospheres containing metal
41 vapours [12-14]. To explore the reinforcing potential of these whiskers, an AA6061 alloy
42 powder has been blended with 10% volume of Al₂O₃ whiskers and consolidated by extrusion.
43 Powder metallurgy is especially suitable for producing aluminium alloy matrix composites
44 (AMCs) because it prevents some wettability problems of Al₂O₃ in molten aluminium and
45 deleterious reactions with the Mg atoms of the matrix that may appear during casting routes
46 [15]. The microstructure and tensile properties of the 6061/Al₂O₃w composite have been
47 evaluated at room and high temperature.

48

49 **2. Experiments**

50 The c-axis α -Al₂O₃ whiskers were obtained employing a cold-wall air-tight chamber
51 furnace with alumina linings using 450 g of quartz sand blended with 45 g of Ni powder. A
52 complete explanation of the experimental procedure for the obtaining of the alumina whiskers
53 can be found in (14). VLS Al₂O₃ whiskers morphology was examined by scanning electron

54 microscopy (SEM) and their composition systematically controlled via energy-dispersive X-ray
55 spectroscopy and X-ray diffractometry.

56 Gas atomised 6061 aluminium alloy powder (particle diameter < 50 μm) was blended with
57 10% volume of Al_2O_3 in a planetary ball mill at 100 rpm for 2.5 hours with a ratio of balls to
58 material of 7:1. The blend was consolidated by extrusion at 723 K, ram speed of 0.3 mm/s and
59 extrusion ratio of 27:1. A monolith 6061 alloy bar was also processed from the alloy powder for
60 comparison purposes.

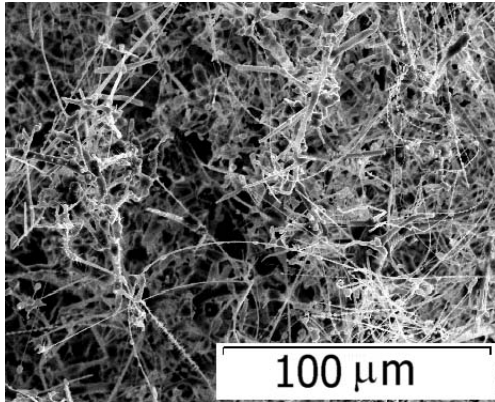
61 The consolidated materials were solution heat treated at 793 K for 0.5 hours and water
62 quenched. Cylindrical tensile specimens of 3 mm diameter and 20 mm gauge length were
63 employed to perform tensile tests on T6 specimens (maximum hardness). Yield stress ($\sigma_{0.2}$),
64 ultimate tensile strength (UTS) and elongation to fracture (ϵ) were determined at room
65 temperature (RT) and at 373 K, 473 K and 573 K at a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$. Elastic modulus
66 was measured on cylindrical samples of 6 mm in diameter and 40 mm in length according to
67 ASTM E1876 and C1259. Microstructural characterization was performed by SEM with a FEG
68 JEOL 6500. Image analysis of the composite was performed on backscattered electron images
69 and data treated with Image-Pro Plus software.

70

71 **3. Results and discussion**

72 Figure 1 shows a SEM micrograph of a bundle of the as-produced VLS c-axis $\alpha\text{-Al}_2\text{O}_3$
73 whiskers that can exhibit lengths of hundreds of microns with aspect ratios that can be larger
74 than 100 and a high level of size dispersion. In the bundles, apart from alpha-alumina, traces of
75 Ni, Fe, Si and SiO_2 were detected as extrinsic contamination.

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77

78 Fig. 1. VLS Al₂O₃ whiskers.

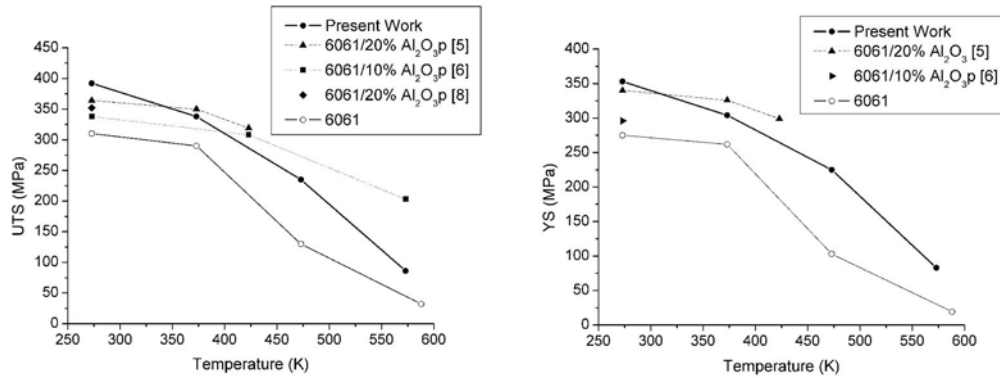
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80 The 6061/Al₂O₃w extruded composite show no agglomerates and no band structure,
81 typically found in extrusion of powders. The Al₂O₃ whiskers appear shorter in average
82 indicating that they became broken during the processing. Image analysis revealed that the
83 composite presents about 15% volume of Al₂O₃ whiskers, i.e. aspect ratio above 5, which are
84 typically between 10 and 20 μm long and 2 to 4 μm in diameter. The remaining Al₂O₃ show an
85 aparent aspect ratio below 5 and diameter of about 2 μm. Whiskers are aligned along the
86 extrusion direction, so that 90% of them have their major axis inside an angle of less than 20°
87 around this direction. This alignment occurred during the extrusion [16]. Apart from the Al₂O₃
88 whiskers, three types of particles appear as contamination from the alumina whiskers production
89 process: Ni(Fe)-rich, Si-rich and Si- and O-rich particles. These will be described below in more
90 detail.

91 In comparison with the unreinforced alloy, the 6061/Al₂O₃w composite presents
92 significantly higher tensile (and specific properties) for the whole temperature range, Figure 2.
93 Comparison with 6061 matrix composites reinforced with Al₂O₃ particles [5-7] shows the
94 considerable advantage of Al₂O₃w, even when comparing with composites reinforced with 20%
95 volume of particles [5, 7], while keeping an ε of 4%. In contrast, when test temperature is
96 increased, tensile strength diminished more abruptly than in the other composites. On the other
97 hand, elastic modulus of 6061/10%Al₂O₃w resulted in 90 GPa, higher than those of other 10%

98 volume Al_2O_3 reinforced composites [4, 7]. Moreover, specific elastic modulus of the composite
99 is about 20% higher than that of the alloy matrix, i.e. 32×10^6 against 26×10^6 Nm/kg.

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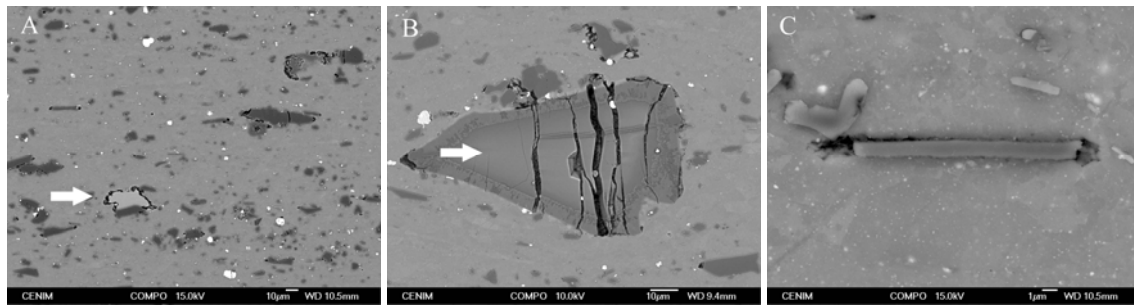
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102 Fig. 2. UTS and $\sigma_{0.2}$ of 6061/10% Al_2O_3 w, 6061 and comparable composites at different temperatures.

103

104 Figure 3a and b show backscattered images of longitudinal sections close to the fracture
105 surface of specimens tested at RT. In addition to Al_2O_3 , three types of particles are present. A
106 first one appears brighter and is Ni-rich, with some Fe occasionally also being found. These
107 particles are quite spherical in shape and of less than 5 μm in diameter. A second type, arrowed
108 in Figure 3a, contains only Si, is bright-grey, irregular in shape and up to 30 μm in size. Finally,
109 a third type, arrowed in Figure 3b, corresponds to SiO_2 . These particles are very large
110 polyhedrals (typically of about 100 μm in diameter) and are surrounded by a Mg and O-rich
111 phase with Al_2O_3 whiskers sometimes embedded. Under RT tensile conditions the Al_2O_3
112 whiskers become mainly cracked, which indicates that the shear strength at the 6061
113 matrix/ Al_2O_3 interface was higher than the Al_2O_3 whisker fracture strength, and thus, that a
114 strong mechanical bond between matrix and reinforcement has been generated during the
115 extrusion process [17]. With regards to the contaminating particles, those that are Ni-rich seem
116 not to be affected during the tensile test so that most of them appear neither cracked nor
117 debonded. In contrast, the Si particles appear mostly debonded from the matrix, whereas the
118 SiO_2 particles appear catastrophically broken, with cracks running through them perpendicular

119 to the loading direction. The fracture mechanism clearly changes at high temperature, as it is
120 shown in the micrograph of Figure 3c. In this case, decohesion becomes the main cause of
121 damage, so that void nucleation can clearly be observed at whisker and contaminant
122 particle/matrix interfaces.
123



124
125 Fig. 3. Longitudinal sections close to the fracture surface of specimens tested at (a) and (b) RT, (c) 573 K.
126

127 The high RT tensile and specific tensile properties of the 6061/Al₂O₃w composite, even
128 though it contains contaminating particles and even though the Al₂O₃w became broken during
129 processing, validates the quality of the VLS alumina whiskers and the suitability of the
130 fabrication route that promotes a strong bonding between matrix and reinforcement.
131 Comparison with similar composites reinforced with Al₂O₃ short fibres or SiC whiskers is not
132 straightforward because few results are available with the same matrix in T6. Composites with
133 higher volume fraction of fibres or whiskers present, obviously, higher properties. For example,
134 RT $\sigma_{0.2}$ and UTS of a 6061 alloy reinforced with 18% volume of short Al₂O₃ fibres [8] were 400
135 MPa and 470 MPa, respectively, and for 6061 reinforced with 20%SiCw [18, 19] were 440 MPa
136 and 500 MPa, respectively, with similar UTS reported for a 6061/22%SiCw composite [20]. In
137 all these materials, not only was reinforcement volume fraction about two times larger than in
138 our composite material, but also median Al₂O₃w aspect ratios were as high as 100. This
139 indicates that improved properties can be achieved for the 6061/Al₂O₃w composite by
140 optimising the blending step, so that the large aspect ratio of the as-produced VLS Al₂O₃
141 whiskers can be preserved. Moreover, further improvement should be obtained if a cleaning

142 protocol is applied to the Al₂O₃ bundles in order to eliminate the weakly adhered Si particles
143 and the brittle SiO₂ ones.

144

145 **4. Conclusions**

146 VLS α -Al₂O₃ whiskers have been investigated as reinforcement in a powder metallurgy
147 6061/10% vol. Al₂O₃w composite. In comparison with the unreinforced alloy, the 6061/Al₂O₃w
148 composite presents significantly higher mechanical properties and specific properties at room
149 and high temperatures. At RT, comparison with 6061 matrix composites reinforced with Al₂O₃
150 particles shows the advantage of Al₂O₃w reinforcement, even when comparing with composites
151 reinforced with 20% volume of particles. This is attributed to the high quality of the Al₂O₃
152 single crystals obtained by the VLS process and to the strong bonding between them and the
153 6061 alloy matrix attained during consolidation. Breaking of the whiskers occurred during
154 processing. Three types of contaminating particles are present, coming from the VLS process:
155 Ni(Fe)-rich, Si and SiO₂ particles. The first type did not affect tensile properties, whereas the
156 other two clearly produced a deleterious effect. Improved properties are expected by enhancing
157 the purity of the Al₂O₃w bundles and by preserving the high whiskers aspect ratio through
158 optimisation of the blending parameters.

159

160 **Acknowledgements**

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164 **References**

165 [1] V. Lavaste, J. Besson, M. Berger, A.R. Bunsel, J. Am. Ceram. Soc. 78 (1995) 3081.

166 [2] G. Das, Ceram. Eng. Sci. Proc. 16 (1995) 977.

167 [3] T.F. Cooke, J. Am. Ceram. Soc. 74 (1991) 2959.

- 168 [4] B.G. Park, A.G. Crosky, A.K. Hellier, J. Mater. Sci. 36 (2001) 2417.
- 169 [5] L. Ceschini, A. Morri, R. Cocomazzi, E. Troiani, Mat –wiss U Werkstofftech. 34 (2003) 370.
- 170 [6] Chia-Chaw Perng, Jiun-Ren Hwang, Ji-Liang Doong, Mater. Sci. Eng. A171 (1993) 213.
- 171 [7] http://mmc-assess.tuwien.ac.at/data/prm/duralcan/aa6061_al2o3.htm.
- 172 [8] M. Vedani, E. Gariboldi, Acta Metall. 44 (1996) 3077.
- 173 [9] G. Requena, H.P. Degischer, Mater. Sci. Eng. A420 (2006) 265.
- 174 [10] R. Tavangar, L. Weber, A. Mortensen, Mater. Sci. Eng. A395 (2005) 27.
- 175 [11] H. Akbulut, M. Durman, Mater. Sci. Eng. A262 (1999) 214.
- 176 [12] V. Valcárcel, A. Souto, F. Guitián, Adv Mater. 10 (1998) 138.
- 177 [13] V. Valcárcel, A. Pérez, M. Cyrklaff, F. Guitián, Adv. Mater. 10 (1998) 1370.
- 178 [14] V. Valcárcel, C. Cerecedo, F. Guitián, J. Am. Ceram. Soc. 86 (2003) 1683.
- 179 [15] T.P.D. Rajan, R.M. Pillai, B.C. Pai, J. Mater. Sci. 33 (1998) 3491.
- 180 [16] A. Borrego, J. Ibáñez, V. López, M. Lieblich, G. González-Doncel, Scripta Metall. Mater.
- 181 34 (1996) 471.
- 182 [17] V.V. Ganesh, N. Chawla, Mater. Sci. Eng. A391 (2005) 342.
- 183 [18] V.C. Nardone, K.M. Prewo, Scripta Metall. 20 (1986) 43.
- 184 [19] R. B. Bhagat, M.B. House, Mater. Sci. Eng. A144 (1991) 319.
- 185 [20] Z.Y. Ma, C.K. Yao, Mater. Chem. Physics. 25 (1990) 463.

186

187 **Figure Captions**

188

189 Figure 1. VLS Al₂O₃ whiskers.

190

191 Figure 2. UTS and $\sigma_{0.2}$ of 6061/10%Al₂O₃w, 6061 and comparable composites at different
192 temperatures.

193

194 Figure 3. Longitudinal sections close to the fracture surface of specimens tested at (a) and (b)
195 RT, (c) 573 K.