Polskie Towarzystwo
Materiałów Kompozytowych
Polish Society of
Composite Materials

Kompozyty 11: 2 (2011) 142-146

KOMPOZYTY
COMPOSITES

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Otrzymano (Received) 09.02.2011

MICROSTRUCTURE AND PROPERTIES OF 7475 ALUMINIUM ALLOY MATRIX NANO-COMPOSITES WITH 10-20% Al_2O_3 OR AlN ADDITIONS

The microstructure and properties of two types of 7475 aluminium alloy matrix composites - with additions of 10÷20 wt.% of nano-Al_2O_3 or AlN (<40 μm) were investigated. The composites were produced through powder-metallurgy processing. Pre-alloyed 7475 aluminium powders were mixed with ceramic particles and milled in a high energy planetary Fritsch ball mill for up to 40 hours. Next, they were compacted at 380°C and 600 MPa. The microstructure of the obtained composites was studied using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The performed investigations proved that both types of composites show a good dispersion of ceramic phases. The composite matrix was characterized by a fine grain size, i.e. less than 100 nm and contained a high density of intermetallic, Zn, Cu or Fe rich phases. The EDX chemical analysis indicated the local presence of an MgO phase at the metal/Al_2O_3 and metal/AlN interfaces. The microhardness of the compacts were in the range of 315÷355 HV and 250÷280 HV range for composites with micro-AlN or nano-Al_2O_3, respectively. It indicates that the size of the nano-crystalline matrix and intermetalic precipitates play a more important role than that of the reinforcing ceramic particles.

Keywords: nanocomposites, metal matrix composites, nano-crystalline aluminium alloy matrix, 7475/AlN, 7475/Al_2O_3

MIKROSTRUKTURA I WŁAŚCIWOŚCI NANOKOMPOZYTÓW O OSNOWIE ZE STOPU ALUMINIUM 7475 Z DODATKIEM 10-20% Al_2O_3 LUB AlN

Kompozyty o osnowie ze stopów aluminium są grupą materiałów, które łączą w sobie charakterystyczną dla metali dobrą plastyczność z dużą wytrzymałością i wysokim modułem sprężystości ceramiki. Wśród tych materiałów szczególnie ciekawe wydają się być kompozyty o nanokrystalicznej osnowie, dającej możliwość dodatkowej poprawy własności mechanicznych.

W niniejszej pracy przedstawiono wyniki obserwacji mikrostruktury i pomiarów twardości dwóch typów nanokompozytów o osnowie ze stopu aluminium 7475, tj. z dodatkiem 10÷20% wagowych nano-Al_2O_3 lub mikro-AlN. Kompozyty wytworzono metodą metalurgii proszków. Proszek stopu AA7475 zmieszano z cząstkami ceramicznymi w ilościach 10 i 20% wagowych, a następnie poddano mieleniu w wysokoenergetycznym młynku kulowym marki Fritsch przez okres 40 godzin. Zastosowano prędkość obrotów 200 obr/min i stosunek wagowy kul stalowych do proszku 10:1. Następnie, proszki sprasowano pod ciśnieniem 600 MPa w umieszczonej w próżni 10^{-2} bar matrycy podgrzewanej za pomocą generatora wysokiej częstotliwości do 380°C. Mikrostruktura kompozytów została zbadana za pomocą skaningowej mikroskopii elektronowej (SEM) oraz transmisyjnej mikroskopii elektronowej (TEM). Wykonane zostały również badania mikrotwardości Vickersa wytworzonych kompozytów.

Przeprowadzone obserwacje mikrostruktury sprasowanych kompozytów wykazały równomierny rozkład cząstek ceramicznych zarówno dla dodatku AlN, jak i Al_2O_3. W skład osnowy kompozytów wchodziły nanokrystaliczne ziarna (~100 nm) oraz międzymetaliczne wytrącenia bogate m.in. w żelazo, miedź, cynk. Lokalnie, widoczne były również obszary o ziarnach osnowy dochodzących do 500 nm, pozbawionych nanokrystalicznej fazy międzymetalicznej. Pustki na styku metalowa osnowa/cząstka ceramiczna były również obserwowane, jednakże całkowita porowatość wynosiła <1%. Pomiary składu chemicznego techniką EDX wskazały na lokalną obecność tlenku magnezu w granicy osnowa/AlN lub Al_2O_3. Pomiary mikrotwardości kompozytów wykazały natomiast, że wzrost frakcji fazy wzmacniającej z 10 do 20% wpływa na wzrost twardości kompozytu z 250 do 280 HV w przypadku Al_2O_3 oraz z 315 HV do 355 HV w przypadku AlN.

Słowa kluczowe: nanokompozyty, kompozyty o osnowie metalowej, 7475/AlN, 7475/Al_2O_3

INTRODUCTION

Aluminium alloy-matrix composites combine the properties of ductility and toughness of metals with the high strength and high modulus of ceramic materials [1--10]. The 7XXX series alloys are characterized by high strength, low density and good plasticity, which make them the material of choice for composite matrix applications [1-5]. Due to composite micro-porosity, their room temperature strength is usually lower than that of hot worked and aged 7XXX alloys, though, they can retain their beneficial properties up to higher temperatures [4]. Additionally, the composites show very good wear resistance [2, 3].

One of the most popular methods of composite production is the powder metallurgy route [1, 5, 7, 9] relying on the mixture of pre-alloyed powders and reinforcement like ceramic nanoparticles [6, 9], nanotubes [11], etc. Other techniques, like casting [3] or spray co-deposition [10] are also applied. Ball milling allows not only very good mixing of powders, but also provides extreme refinement of the grain size and in consequence, significantly improves the mechanical properties of the composite. Therefore, the studies of the effect of this process are one of the purposes of this report.

The application of mechanical alloying to a mixture of 2014 aluminium alloy powder and VC carbides allowed to obtain a composite with uniformly distributed ceramic particles and significant hardening [7]. A similar effect was obtained in aluminium strengthened with Al_2O_3 or SiC [8]. Ball milling was also used to introduce Al_2O_3 nanoparticles into the 6061 alloy, to produce a final composite by hot pressing, which increased wear resistance by 145% in comparison to a conventional aluminium alloy. Occasionally, as high strength as ~1000 MPa can be obtained, as in the case of the composite consisting of the 6061 aluminium alloy and 20% ZrO_2 [9]. Such good mechanical properties were not only due to the addition of nanoparticles, but also due to the grain refinement of the aluminium solid solution obtained during ball milling.

Taking into account the growing interest in the development of nanocrystalline metal matrix composites, in the present work the pre-alloyed powder of high strength AA7475 was ball milled with Al_2O_3 nanoparticles or micro-AlN. Next, they were hot pressed at a temperature below the fast grain growth range. Finally, the microstructure of the obtained material was characterized using scanning and transmission electron microscopy.

EXPERIMENTAL PROCEDURE

The powder from the 7475 aluminium alloy (5.7% Zn, 2.2% Mg, 0.7% Fe, 1.6% Cu, 0.1% Mn, 0.5% Zr- rest Al, Hydro Aluminium Company) was mixed with nano-size α-Al_2O_3 or −325 mesh AlN powder (Alfa Aesar). In both cases 10 wt.% and 20 wt.% of the ceramic phases were applied. Then, the powders were subjected to high energy ball milling in the planetary Fritsch mill Pulverisette 5 for 40 hours in tool steel containers filled with argon. The rotational speed of 200 rpm and ball-to-powder weight ratio of 10:1 were utilized. The powders were compacted in a VEB 40 hydraulic uniaxial press in a mould placed in a vacuum and heated using a high frequency generator. Discs about 5 mm thick, 20 mm in diameter were obtained after hot pressing in a vacuum of 10^{-2} bar, at a pressure of 600 MPa and temperature of 380°C. The microstructure of the hot compacted composites was characterized using an FEI Quanta 3D scanning electron microscope and a Tecnai G2 F20 (200 kV) transmission electron microscope equipped with an integrated EDAX system. Thin foils for TEM investigations were prepared either by mechanical polishing, dimpling with a Gatan Dimple Grinder (Model 656) and finally ion Ar milled with a Leica EM RES101 system. A Quanta 3D focused ion beam (FIB) was also used. Microhardness Vickers measurements were performed using a CSM-Instruments tester.

RESULTS AND DISCUSSION

The microstructure observations of the compacts using scanning microscopy showed that in the A7475/AlN composite, the ceramic particles characterized by slightly darker than the matrix contrast are evenly distributed in the aluminium matrix (Fig. 1a). This composite also contains a high density of very fine intermetallic particles represented by light contrast. The above identification was confirmed by using the EDS technique, which beside the aluminium and nitrogen in the AlN, allowed confirmation of the presence of Zn, Cu, Mg, Fe in the intermetallics. The occasional presence of voids of a size comparable with the intermetallics is indicated by the darkest features. The AA7475/Al_2O_3 composite microstructure is also characterized by the presence of intermetallic precipitates and voids, while the ceramic particles are practically indistinguishable due to a similar contrast to the matrix, i.e. nearly the same average atomic mass value (Fig. 1b).

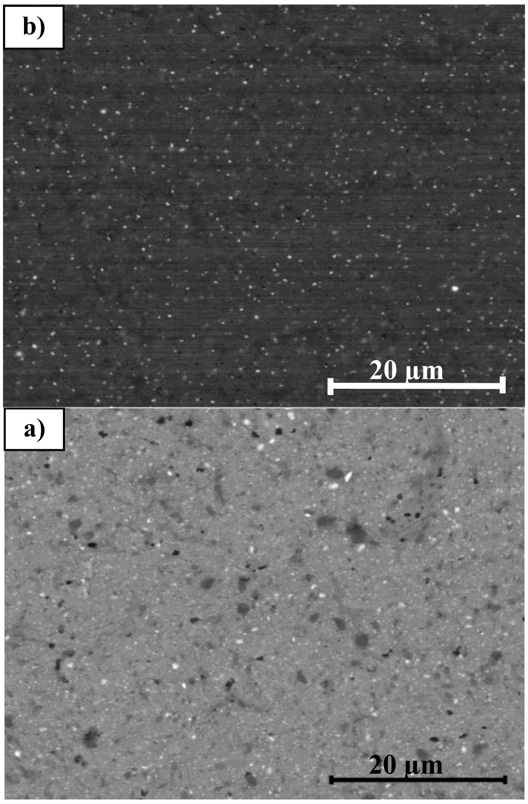

Fig. 1. SEM (BSE) image of microstructure of: a) 7475/$AlN_{10\%}$ and b) 7475/$Al_2O_{3\ 10\%}$ composites

Rys. 1. Obrazy mikrostruktury SEM (BSE) kompozytów: a) 7475/$AlN_{10\%}$, b) 7475/$Al_2O_{3\ 10\%}$

The quantitative measurements performed on the SEM micrographs indicated that the average crystallite size of intermetallic precipitates was 200 nm and 120 nm for 7475/AlN and 7475/Al_2O_3, respectively. Moreover, in the case of the latter composite, the maximum size of voids was also smaller, namely below 400 nm compared to 800 nm in the 7475/AlN case (Fig. 2). The intermetallic crystallite size may be overestimated as compared to TEM observations due to contrast forming principles.

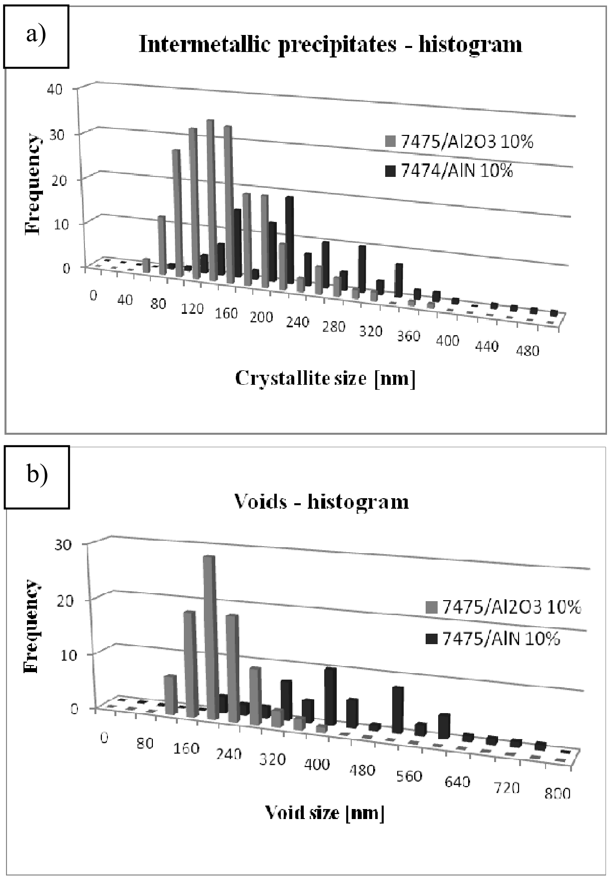

Fig. 2. Size distribution of: a) intermetallic crystallites and, b) voids in 7475/$AlN_{10\%}$ and 7475/$Al_2O_{3\ 10\%}$ composites

Rys. 2. Rozkład rozmiaru: a) międzymetalicznych krystalitów, b) pustek w kompozytach 7475/$AlN_{10\%}$ i 7475/$Al_2O_{3\ 10\%}$

The accompanying transmission electron microscopy observations not only confirmed the nano-crystalline nature of the intermetallics, but also showed that the matrix is characterized by a similarly small "nano-size", i.e. about 100 nm. The AlN particles were easily distinguished due to their large size and high dislocation density (Fig. 3a). Parts of the interfaces of the matrix/AlN particles showed the presence of porosity, as elucidated by whitish fringes. Along with the SEM investigation also in the TEM, in the case of the nano-crystalline Al_2O_3 particles their close to matrix average - mass contrast prevented their identification using the bright field (BF) imaging mode (Fig. 3b). Occasionally, areas with larger matrix grains, i.e. up to about 400 nm, devoid of an intermetallic phase were also observed.

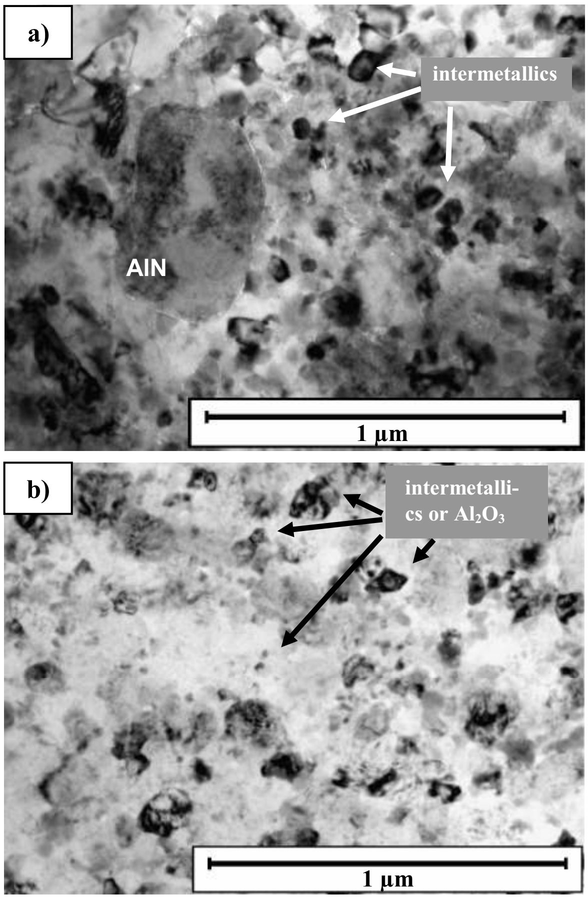

Fig. 3. Bright-field TEM micrographs of: a) 7475/$AlN_{10\%}$, and b) 7475/$Al_2O_{3\ 20\%}$ composites

Rys. 3. Obrazy TEM w jasnym polu kompozytów: a) 7475/$AlN_{10\%}$, b) 7475/$Al_2O_{3\ 20\%}$

The scanning transmission electron microscopy observations using the High Angle Annular Dark Field (HAADF) detector backed by local chemical analysis with the EDS technique eventually enabled identification not only of the AlN but also the very fine Al_2O_3 particles. The latter were mostly of a spherical shape and homogenously distributed throughout the matrix. Very fine Mg and O rich particles were attached to both the AlN and Al_2O_3 strengthening phases (Fig. 4). Their presence at the Al_2O_3 may indicate that it is an MgO phase formed by a reaction of Al_2O_3 with magnesium from the metallic solid solution. However, in the case of AlN only the surface oxide layer present at every material may serve as the source of oxygen for MgO formation. Beside the ceramic particles are smaller and brighter particles which are the previously discussed intermetallics containing Al, Cu and Fe, i.e. possibly phases from the Al - Cu - Fe system.

The microhardness measurements performed on the AA7475/AlN and AA7475/Al_2O_3 composites indicate, that increasing the amount of reinforcing particles addition from 10 to 20 vol.% helps to raise the hardness from 250 to 285 HV and from 315 to 355 HV for the former and the latter, respectively (Fig. 5). These results are much higher than those for composites with only 5% ceramic [1, 5].

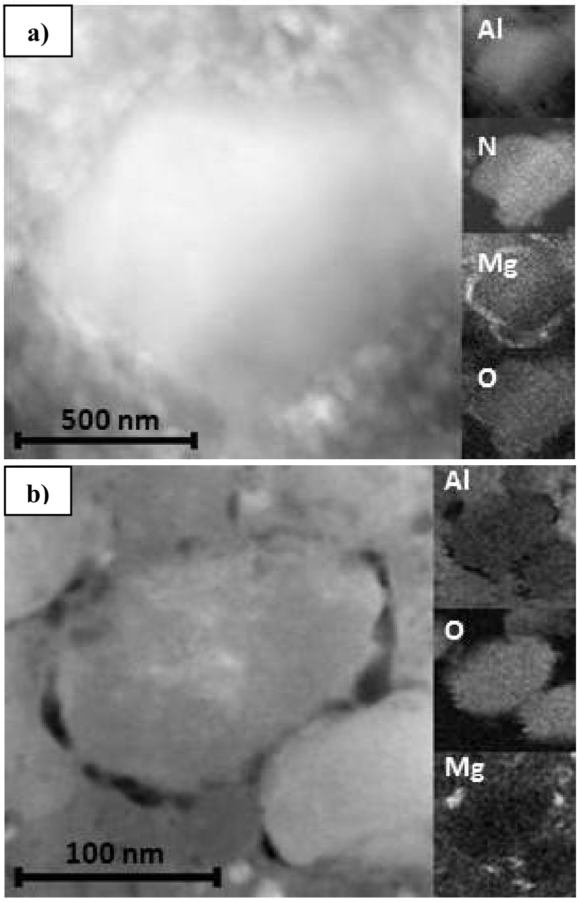

Fig. 4. HAADF STEM micrographs of: a) 7475/$AlN_{20\%}$, b) 7475/Al_2O_3 $_{20\%}$ composites

Rys. 4. Obrazy HAADF STEM kompozytów: a) 7475/$AlN_{20\%}$, b) 7475/Al_2O_3 $_{20\%}$

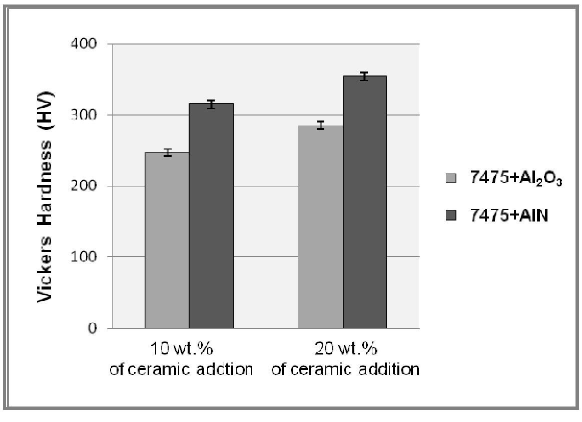

Fig. 5. Vickers micro-hardness of 7475/Al_2O_3 and 7475/AlN composites

Rys. 5. Mikrotwardość Vickersa kompozytów 7475/Al_2O_3 i 7475/AlN

The properties of the latter could be lowered also because the free sintering applied in their case which may result in higher porosity, as compared with hot pressed sintering for the present compacts. Similar high values were reported for highly densified ball-milled extruded composites of the 2017 alloy matrix with 5÷15% SiC or Al_2O_3 additions [8] and the extruded composite with 15÷20% SiC [10]. It indicates that in the case of composites prepared from powders, their low porosity is very important. An even higher hardness may be achieved by additional ageing of the metal matrix, as shown in [10]. In the case of the present study, the higher strength of the aluminum alloy matrix was attained by strong refining of the grain size and formation of even finer intermetallic precipitates. Generally, the amount and size of the ceramic phase addition can be considered as only one of the factors influencing the strength of composites [1, 5, 8, 10], which is also supported by the present study.

The higher hardness of composites reinforced with micro- rather than with nano-particles as compared to the AA7475 alloy itself (which hardness in T6 condition equals ~173 HV), indicates that the intermetallics and matrix grain size play a very important, if not decisive role in the material hardening mechanism.

SUMMARY

The performed investigations proved that both types of composites show a good dispersion of ceramic phases independent of the differences in their size or amount of the added strengthening phase. Hot pressing in a vacuum of ball-milled 7475 alumnium alloy powder with ceramic additions of Al_2O_3 or AlN powders allowed us to obtain composites of a nanocrystalline matrix with a grain size of <100 nm containing a high density of even finer intermetallic, Zn, Cu or Fe rich particles. The local chemical analysis with EDX indicated occasional presence of an MgO phase at the interfaces of both types of composites. The produced composites showed significant hardness improvement over that of the matrix, i.e. 250÷285 HV for the Al_2O_3 addition and 315÷355 HV for the AlN addition.

Acknowledgements

The work was supported through "KomCerMet" project (no. POIG.01.03.01-14-013/08-00) in the framework of the Operational Programme for Innovative Economy, as well as project no. POKL. 04.01.00-00-004/10 co-financed by the European Union within the European Social Fund and Development Project no. OR00000508.

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