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COMPOSITES THEORY AND PRACTICE

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MICROSTRUCTURE AND MECHANICAL PROPERTIES OF AA7475/AlN COMPACTS WITH VARIED REINFORCING PARTICLES SIZE

The microstructure and properties of 7475 aluminium alloy matrix composites - with additions of 10 wt. % of AlN powders of different particle size: <40 µm, ~1 µm and <1 µm were investigated. The composites were produced by means of powder-metallurgy. Pre-alloyed 7475 aluminium powders were milled with ceramic particles in a high energy planetary Fritsch ball mill for up to 40 hours and subsequently vacuum hot pressed at 380°C and 600 MPa. The microstructure of the obtained composites was studied using a metallographic microscope and scanning electron microscope (SEM). The performed investigations indicated good dispersion of ceramic phases. However, in the composite with the submicron AlN addition, a small tendency for agglomeration occurred. All of the composites were characterized by high density of intermetallic, Zn, Cu, Mg or Fe rich phases. Recrystallized Al grains were observed in the composite with the submicron AlN addition, indicating that the fine particles did not retard grain boundaries movement. The hardness of the consolidated samples was highest for the ~1 µm ceramic powder addition - nearly 320 HV in comparison to 250 HV for the finer powder addition. The compression tests showed 830 MPa of ultimate compression strength of the samples with the ~1 µm AlN particles, which was slightly higher than that with the submicron particles addition. The higher strength of the composites reinforced with the micro- rather than with submicro-particles suggests that the size of the ceramic phase addition can be considered as only one of the factors influencing the composite strength.

Keywords: metal matrix composites, aluminium alloy matrix, 7475/AlN, powder metallurgy

MIKROSTRUKTURA I WŁASNOŚCI MECHANICZNE WYPRASEK AA7475/AlN O RÓŻNEJ WIELKOŚCI CZĄSTEK ZBROJĄCYCH

Kompozyty o osnowie ze stopów aluminium zbrojone dyspersyjnie cząstkami ceramicznymi są grupą materiałów charakteryzującą się kombinacją dobrej plastyczności, niskiej gęstości, wysokiej wytrzymałości właściwej oraz odporności na kruche pękanie, która sprawia, że są szeroko stosowane w przemyśle lotniczym i motoryzacyjnym. Mimo wieloletniego już zainteresowania tego typu kompozytami, wciąż podejmowane są próby poprawy właściwości istniejących kompozytów oraz projektowania nowych w celu osiągnięcia konkretnych pożądanych własności. Jedną z możliwych dróg poprawy własności kompozytów jest zmniejszenie wielkości zarówno ziaren osnowy, jak również fazy zbrojącej. W niniejszej pracy przedstawiono wyniki obserwacji mikrostruktury i pomiarów wytrzymałości nanokompozytów o osnowie ze stopu aluminium 7475, tj. z dodatkiem 10% wagowych AlN o trzech różnych wielkościach cząstek: <40 µm, ~1 µm i <1 µm. Kompozyty wytworzono metodą metalurgii proszków. Proszek stopu AA7475 zmieszano z cząstkami ceramicznymi w ilości 10% 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 mikroskopu metalograficznego oraz skaningowego mikroskopu elektronowego (SEM). Wytworzone kompozyty poddane zostały badaniu twardości metodą Vickersa oraz próbie ściskania. Przeprowadzone obserwacje mikrostruktury sprasowanych kompozytów wykazały równomierny rozkład cząstek ceramicznych dla dodatków < 40 µm oraz ~1 µm AlN. Natomiast w przypadku submikronowego dodatku AlN zaobserwowano tendencję cząstek do tworzenia aglomeratów. Osnowa wszystkich wytworzonych kompozytów bogata była w międzymetaliczne wytrącenia zawierające m.in. żelazo, miedź, cynk. Pustki na styku metalowa osnowa/cząstka ceramiczna były również obserwowane, jednakże całkowita porowatość wynosiła <1%. W kompozycie zbrojonym submikronowymi cząstkami AlN widoczne były również obszary o zrekrystalizowanych ziarnach osnowy, pozbawione fazy międzymetalicznej. Pomiary twardości kompozytów wykazały najwyższą twardość kompozytu z dodatkiem ~1 µm AlN - 320 HV - w porównaniu z twardością 250 HV w przypadku dodatku < 1 µm AlN. Najwyższa wytrzymałość na ściskanie została również zarejestrowana dla kompozytu o zbrojeniu ~1 µm, jednakże była tylko nieznacznie większa od kompozytu z dodatkiem submikronowego AlN.

Słowa kluczowe: kompozyty o osnowie metalowej, osnowa ze stopu aluminium, 7475/AlN, metalurgia proszków

INTRODUCTION

Aluminium alloy matrix composites reinforced with ceramic phases present a combination of good ductility, low density, high specific strength and fracture toughness that makes them very useful in automotive and aerospace applications [1-8]. Nevertheless, constant efforts are being made to improve existing composites and to design new ones in order to achieve the desired specific combinations of properties.

One of the possible approaches to improve composite properties is a reduction of both the matrix grain size and reinforcing particle size. Nano-crystalline matrices strengthened by micro-sized or nano-sized reinforcement are expected to have much better microstructure stability and performance than conventional metal matrix composites because of the competition between strengthening by the grain-boundary and particle reinforcement [1]. However, the main requirement for the superior performance of a composite material is homogeneous distribution of the reinforcing phase. In discontinuously reinforced composites, the presence of reinforcement agglomerates significantly deteriorates the mechanical properties [2]. Therefore, a decrease in the reinforcement particle size opens only a theoretical possibility of improving mechanical strength as the tendency for particle clustering may deteriorate the composite final properties [2, 3].

Among discontinuously reinforced metal matrix composites (MMCs), most of the attention has been so far paid to SiC and Al_2O_3 reinforced aluminium composites, which are now well developed and commercialized [3-6]. In recent years, however, aluminium nitride has also gained considerable interest. According to previous research, AlN with its good physical-chemical, mechanical and thermal properties, can enhance the modulus, strength, hardness, wear resistance and high temperature performance of an aluminium alloy matrix [1, 2, 7-9]. What distinguishes aluminium nitride from reinforcing phases commonly applied in metal matrix composites is its good bonding to an aluminium matrix, higher wettability in aluminium, as well as stability at the aluminium/aluminium nitride interface at elevated temperatures [7].

The powder metallurgy route employing mechanical alloying during high energy ball milling is found to be one of the most economical in MMCs production, and, above all, potentially suitable for the fabrication of composites providing a nanometric matrix and particle distribution homogeneity [1, 2, 10]. The application of mechanical alloying to aluminium strengthened with Al_2O_3 or SiC allowed researchers to obtain a composite with uniformly distributed ceramic particles and significant improvement in strength [5, 6]. Occasionally, as high a strength as ~1 GPa can be obtained, as in the case of the AA6061/ ZrO_2 composite [11]. Such good mechanical properties were achieved both due to the addition of nanoparticles and grain refinement of the aluminium solid solution obtained during ball milling.

The main goal of this article is an attempt to fabricate a composite consisting of a fine grain aluminium alloy matrix with fairly uniform distribution of AlN reinforcement differing in size. Therefore, a prealloyed 7475 aluminium alloy powder was ball milled with an addition of aluminium nitride powders and subsequently hot pressed in a vacuum. To produce the composites, three types of AlN powders of different particle size were used: < 40 μm, ~1 μm and < 1 μm in order to compare the strengthening effect.

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 a-325 mesh AlN powder (Alfa Aesar), ~1 μm AlN powder (Hongwu Nanometer) and submicron AlN powder (NaBond Technologies). In all the cases, 10 wt.% of the ceramic phases was applied. Then the powders were subjected to high energy ball milling in a 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 of 10^{-2} bar and heated using a high frequency generator. Discs about 5 mm thick, 20 mm in diameter were obtained after hot pressing at a pressure of 600 MPa and temperature of 380°C. The microstructure of the hot compacted composites was characterized using a Leica DM IRM metallographic microscope and FEI Quanta 3D scanning electron microscope with an integrated EDAX system. Vickers hardness measurements were performed using a Zwick Universal Hardness Tester and compression tests were conducted using INSTRON Model 3382 mechanical testing equipment.

RESULTS AND DISCUSSION

Figure 1 shows TEM microstructures taken from samples with three types of AlN powders used as a composite reinforcing phase. According to the measurements performed on the microcgraphs, the average size of crystallites was estimated at about 6 μm (Alfa Aesar), ~1 μm (Hongwu Nanometer) and ~0,3 μm microfraction with a ~50 nm nanofraction (NaBond Technologies).

Figure 2 shows the optical microstructures of hot pressed composite powders containing 10% of three different AlN additions. In the first two micrographs (a,b), some darker dots can be seen which indicate uniform distribution of the aluminium nitride particles. In the case of the submicron AlN reinforcement (c), the small size of the ceramic particles makes it impossible to distinguish them from the aluminium alloy matrix. The striped arrows indicate porosity in the composite microstructure. The pores are, however, small and relatively infrequent, estimated at less than 1%. In Figure 2c, some areas of lower density of the precipitates (white arrows) are also visible. They are most probably caused by a recrystallization process occurring during hot pressing. It indicates that finer particles did not retard grain boundaries movement and were pushed ahead of the recrystallization front.

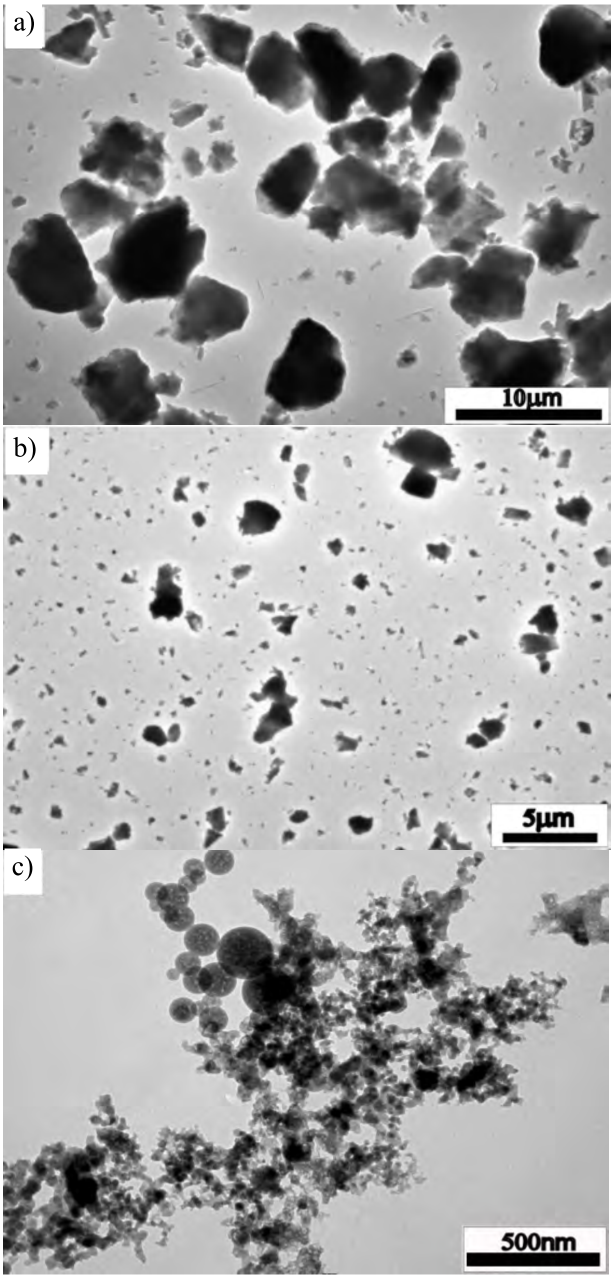

Fig. 1. Bright-field TEM micrographs of AlN powders: a) −325 mesh AlN, b) ~1 µm AlN and c) submicron AlN

Rys. 1. Obrazy TEM w jasnym polu proszków AlN: a) −325 mesh AlN, b) ~1 µm AlN, c) submikronowy AlN

The microstructure observations of the compacts using scanning electron microscopy (Fig. 3) showed that in the composite with the < 40 µm and ~1 µm AlN addition, the ceramic particles (indicated by black arrows) are evenly distributed in the aluminium matrix with no tendency to form agglomerates probably due to mechanical alloying of the metallic and ceramic powders. However, in the composite with the submicron AlN addition, some agglomerations occur (black arrow) but most of the ceramic particles are practically indistinguishable due to their small size and similar contrast to the matrix (Fig. 3c).

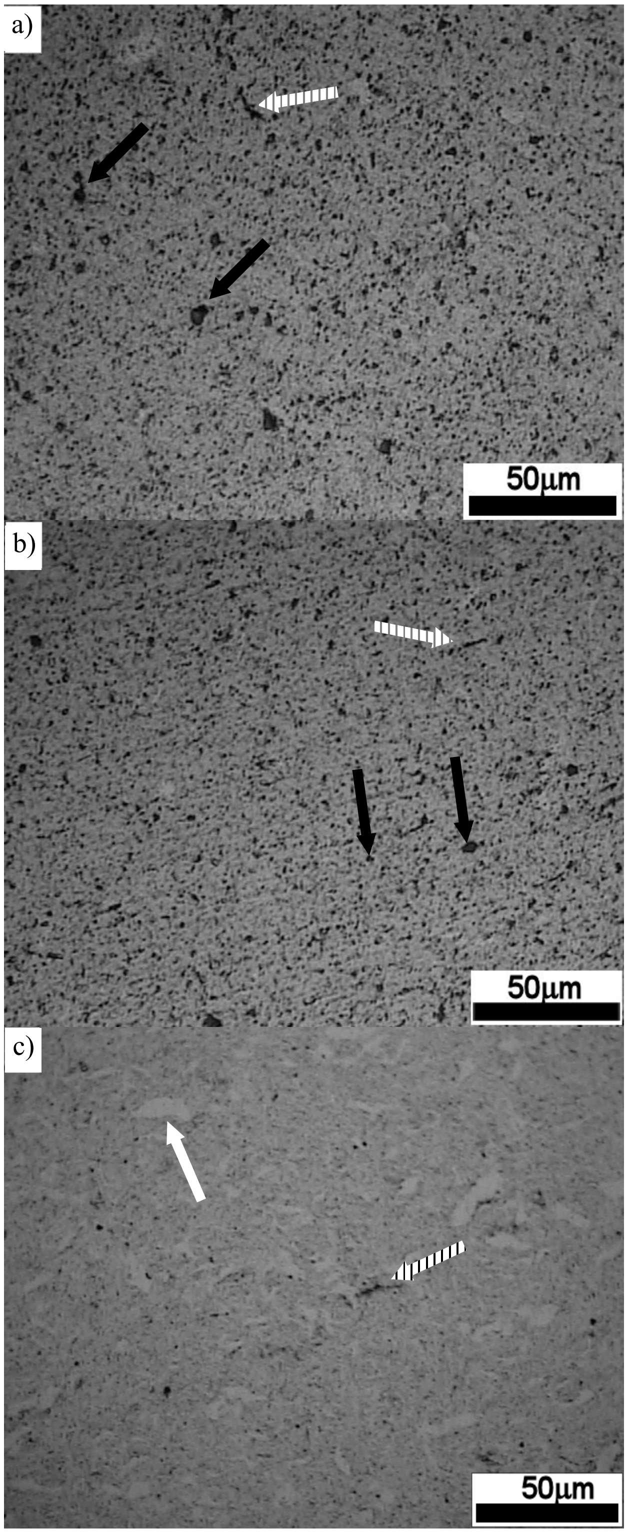

Fig. 2. Optical microstructure of hot pressed composite powders with 10 wt.% of: a) −325 mesh AlN, b) ~1 µm AlN and c) submicron AlN; arrows designation: black - AlN particles, white - recrystallized Al, striped - pores

Rys. 2. Mikrostruktura sprasowanych proszków kompozytowych o 10% wagowych: a) −325 mesh AlN, b) ~1 µm AlN and c) submikronowy AlN; oznaczenie strzałek: czarne - cząstki AlN, białe - zrekrystalizowane Al, prążkowane - pory

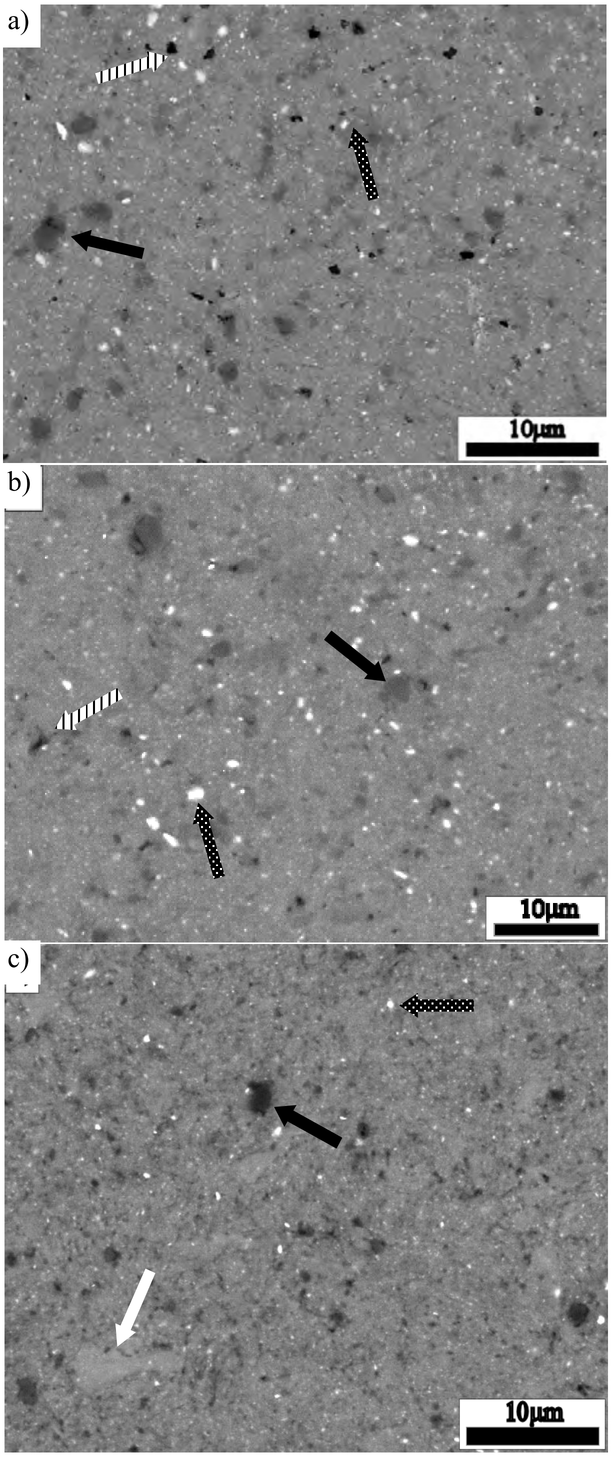

Fig. 3. SEM (BSE) micrographs of hot pressed composite powders with 10 wt.% of: a) −325 mesh AlN, b) ~1 µm AlN and c) submicron AlN; arrows designation: black - AlN particles, white - recrystallized Al, striped - pores, dotted - intermetallic precipitates

Rys. 3. Obrazy SEM (BSE) mikrostruktury sprasowanych proszków kompozytowych o 10% wag. a) −325 mesh AlN, b) ~1 µm AlN i c) submikronowy AlN; oznaczenie strzałek: czarne - cząstki AlN, białe - zrekrystalizowane Al, prążkowane - pory, kropkowane - wytrącenia międzymetaliczne

All the composites also contain a high density of fine intermetallic precipitates (dotted arrows). The identification was confirmed using the EDS technique, which revealed the presence of Zn, Cu, Mg and Fe in the intermetallics. Figure 3 also shows negligible porosity of the composites (striped arrows). The presence of voids is particularly visible in the microstructure of the composite reinforced with the coarser AlN particles (Fig. 3a) in the other two cases the pore content is significantly lower. In Figure 3c some areas of recrystallized aluminium are also visible.

The hardness measurements performed on the three series of AA7475/AlN composites indicate that the addition of 10 wt. % of AlN phase helps to raise the hardness from 240 HV (pure hot-pressed ball-milled 7475 alloy) up to 315 HV in the case of the ~1 µm AlN addition (Fig. 4). The relatively low hardness of the composite reinforced with the submicron AlN particles (250 HV) is probably connected with the recrystallization of the Al matrix and/or AlN particles clustering. When the grain size of the matrix is much bigger than the AlN particles, the Orowan bypass mechanism may operate within those large grains of Al resulting in a lower composite strength. On the other hand, the inhomogeneous microstructure of the composite (agglomerates), makes it prone to easy initiation and propagation of cracks, which cause a decrease in the mechanical properties of composites.

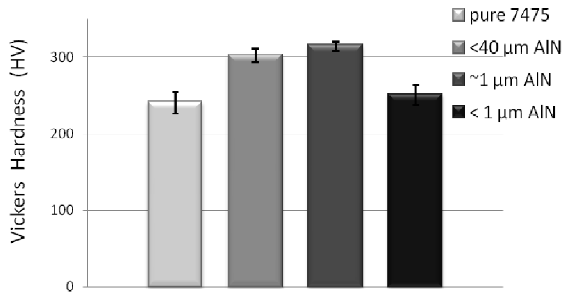

Fig. 4. Vickers hardness of 7475/$AlN_{10\%}$ composites

Rys. 4. Twardość Vickersa kompozytów 7475/$AlN_{10\%}$

Figure 5 shows the stress-strain curves obtained from the compression test of the composite based on 7475 with a 10% AlN reinforcement. The shapes of the presented stress strain curves of all the composites are typical for brittle materials. One can see that the maximum strength - 830 MPa with a deformation of about 6% - corresponds to the ~1 µm AlN reinforcement. The strength of the sample with the submicron size AlN additions is comparable - nearly 820 MPa. It can be explained by the fact that the compression tests show high values of compression strength not only due to ceramic phase additions, but also due to high grain refinement of the 7475 alloy matrix. On the other hand, the compressive strength of the sample with the coarse AlN reinforcement is significantly lower - 550 MPa. The addition of the ceramic phase does not causes a significant increase in the compression strength in comparison to the compression strength of hot-pressed ball-milled 7475 powder, which approaches 700 MPa. This suggests that 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 fine intermetallic precipitates.

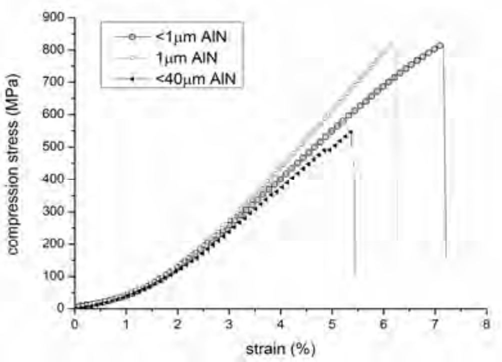

Fig. 5. Compression curve of 7475/AlN$_{10\%}$ composites

Rys. 5. Krzywe ściskania kompozytów 7475/AlN$_{10\%}$

SUMMARY

Hot pressing in vacuum of ball milled 7475 aluminium alloy powder with ceramic additions of AlN allows one to obtain composites with good dispersion of ceramic phases, independent of the differences in their size, containing a high density of fine intermetallic precipitates. The compressive strength of composites with 10% of AlN increase from 650 MPa for hot pressed 7475 milled powder to 830 MPa for a ~1 μm AlN reinforcement. The produced composites showed significant hardness improvement over that of the matrix as well, i.e. 315 HV for the ~1 μm AlN addition. The higher strength of composites reinforced with micro- rather than with submicro- particles suggests that the size of the ceramic phase addition can be considered as only one of the factors influencing the composite strength.

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