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MULTISCALE MICROSTRUCTURE CHARACTERISATION OF METAL MATRIX COMPOSITES (MMC) REINFORCED BY THE ULTRAFINE PARTICLES

This work deals with the explanation, through the multiscale microstructural investigations, the strengthening mechanism in Al/TiC in-situ cast composites. The strengthening effects related to the number and size of TiC particles, grain refinement of the matrix and the lattice mismatch between the Al and TiC crystal lattices were explained, based on two composites containing 5 and 15 wt.% TiC, respectively. The microstructural investigations were supported by tensile test, hardness and abrasive wear resistance. It has been shown that addition of 15 wt.% of TiC particles to the aluminium matrix is more beneficial for mechanical properties and wear resistance than 5 wt.%, even the intensified clustering process of TiC particles.

Keywords: Al/TiC composite; light microscopy; SEM; TEM; mechanical properties; wear

1. Introduction

Metal matrix composites (MMCs) can be produced both by ex situ or in situ [1,2] processes. The main advantages of the second process are: (i) the production of the reinforcement in situ in the molten alloy by a chemical reaction, (ii) good interfacial bonding between the particles and matrix and (iii) the lack of contamination between the matrix and the reinforced phases. This allows to obtain fine and homogeneously distributed particles. Furthermore, this processing method is relatively simple, thus the production can be carried out on a large scale and simply implemented to the industrial scale. Among large number of various MMCs, aluminium-based matrix composites (AMCs) plays an important role due to their low density, high strength-to-weight ratio, excellent corrosion resistance, enhanced wear resistance, favourable high-temperature properties and wide range of applications in automotive, transportation, and defence industries [3-5]. Particulate reinforced composites consist of a uniform distribution of strengthening particles within a matrix. Among several ceramic reinforcing particles including SiC, TiC, B₄C, TiB₂ or Al₂O₃ [6], TiC seems to be the most promising due to its very high melting point (3067°C), high hardness, good thermal and electrical conductivities, and low chemical reactivity [7]. Research on (AMCs) reinforced with TiC particles dates back to

the late 20th century. These studies addressed both manufacturing methods and the resulting microstructure and properties [8-10]. Early efforts primarily centered on producing micrometer-sized particles, often characterized by a nonuniform distribution. Although this approach resulted in improvements to mechanical and tribological properties [11], the degree of enhancement was still unsatisfactory. In the first decade of the 21st century, research focused on improving both particle distribution and particle size (i.e., reducing particle dimensions), as well as identifying and implementing more advanced methods [12-14] to achieve optimal structures and properties of Al-TiC composites [12]. Currently, studies mainly target the optimization of manufacturing technology to maximize the reinforcing effect of the TiC phase. By controlling the reinforcement type, size, morphology and relative quantity the different properties of composite can be tailored [15]. The research on composite materials is mainly focused on the explanation of strengthening mechanism [16-20] that can explain the unique properties of MMCs including high strength and ductility. It has been assumed that the several strengthening mechanism contributions, including: load transfer effect, Hall-Petch strengthening, Orowan strengthening coefficient of thermal expansion (CTE) and elastic modulus (EM) mismatch may occur in composites while in some cases the final mechanical properties of materials are the effect of more

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than one strengthening effect. In order to explain the role of the individual strengthening components, microstructural studies conducted at different scales are important, because each of the mechanism can occur in different conditions and for various sizes of microstructural factors, i.e. grains, particles, interphase boundary, dislocations, crystal lattice. Our previous works concerning in-situ cast TiC/Al or (Ti,W)C/Al composites were especially focused on the description of the influence of TiC and (Ti,W)C particles – their morphology, size, volume fraction and distribution in the matrix as well as the coherency and purity of the particle-matrix interface on the mechanical properties [21–24]. It should be mentioned that strong particle-matrix interface and microstructure design in an atomic scale plays the key role in the development of the AMCs exhibiting high strength-ductility synergy. Yan et al. [25] presented how the properties of composites can be influenced by the manipulation of the interface bonding and microstructure via tuning interfacial reactions. Moreover, another interesting factor allowing for the increase of mechanical properties is applying a dual size particles in in-situ cast AMCs [26]. In this case, both groups of particles in size in the range of 100–300 nm and 10–20 nm should have a uniformed distribution in the matrix controlled by the microscopic investigations with different magnifications. Other microstructural features related to CTE and EM mismatch were observed in SiC/Al composite [27]. Here, the in-situ transmission electron microscopy (TEM) tensile test was conducted to reveal the strengthening behaviour of stacking faults (SFs) during micro-plasticity. The results showed that the extra strengthening effect in SiC/Al composites with SFs includes elastic interactions of SF extension, sessile stair-rod partial dislocations and interactions of SF networks and dislocation arrays. Therefore, it is visible that role of wide scale microstructural analysis in the study of composite materials, in particular to describe the microstructural strengthening effects composites, is extremely important but rarely presented in the literature. In this work, we presents a comprehensive studies of multiscale microstructure characterisation of AMC reinforced by the TiC particles performed from millimetre down to angstrom scale. This allowed for the demonstration of all strengthening effects occurring in the investigated composites and their correlation with the mechanical properties.

2. Experimental procedure

The composites, based on aluminium 1000 alloy (the chemical composition was shown in our previous work [21]) were produced in-situ by Self-propagating High-temperature Synthesis in Bath (SHSB) method. The detailed experimental procedure of in situ cast process including the preparation of the reactants of TiC reaction was presented in Ref. [24]. In this work, we cast two types of TiC/Al composite differing with the weight percentage (wt.%) of TiC reactants 5 and 15 respectively while casting process was conducted without the addition of moderator to control the reaction of TiC synthesis. The aluminium 1000 alloy ingot was melted in an Al₂O₃ crucible in a Balzer's

induction vacuum furnace. Ti-C green compacts prepared from mixed powders of pure Ti and graphite were added into to a crucible on liquid surface of alloy, and then, the suspension of created TiC in molten Al was cast into a metal mould and cooled in air. Finally, we produced Y-shaped castings weighing approximately 370 g. Microstructural studies were started using the KEYENCE VHX 7000 digital microscope allowing for observations with low magnifications. Automatic image stitching of images taken along the whole composite ingots were performed to show the entire ingot morphology. The ingots were mechanically polished using first sand papers with grit from 1000 to 7000, then 1 µm, 1/4 µm diamond suspensions and finally colloidal silica. In order to reveal the grain microstructure, electropolishing and etching was carried out using Barker's reagent with the following parameters: 20V, 60s, -10°C. The images taken at the higher magnifications and chemical composition of the composites were examined by scanning electron microscope (SEM) FEI Quanta 3D FEGSEM equipped with X-ray energy dispersive spectrometer EDAX GEMINI 4000 system. The detailed microstructural characterization including determination of crystal structure and high resolution (HRTEM) observations in the atomic scale was performed by use of transmission electron microscopes (TEM) FEI Tecnai G2 200 kV and Thermo Fisher Titan Themis 200 G3. Samples for TEM in the form of lamellas were prepared by Focused Ion Beam (FIB) method using Thermo Fisher Scios 2. The hardness was measured using United Test hardness tester by applying the Vickers method using a load of 9.8 N (HV1) for 10 s. The abrasive wear resistance of the composite materials was assessed using the ball-on-disc method on an ELB-07 tribotester (ELBIT, Poland) based on ISO 20808:2004(E) standards. The main test parameters were as follows: Al₂O₃ ball diameter of 3 mm, friction track diameter of approximately 3.5 mm, disc rotational speed of 192 rpm and loading force of 4 N. Each test was conducted at room temperature for 10000 seconds. The wear volume measured by Keyence VHX 7000. The mechanical properties of the composites were determined in the tensile tests using an Instron TT-DM machine equipped with an electronic measuring system. Tests were conducted at 293K with an initial strain rate of 10⁻³s⁻¹.

3. Results and discussion

The microstructure observations of the entire composite ingot, taken at low magnification, allows for control the casting process parameters (efficiency of suspension mixing, conglomeration of particles etc.) as well as verification of in-situ formation of TiC particles process and optimisation of their volume fraction and size. Fig. 1 presents the set of micrographs of two ingots with various contents of TiC particles and corresponding images taken in polarized light.

The composite with various content of TiC particles shows a significant differences in microstructure. The material containing 5 wt.% of particles reveals homogeneous distribution of the reinforcement throughout the entire volume while only small

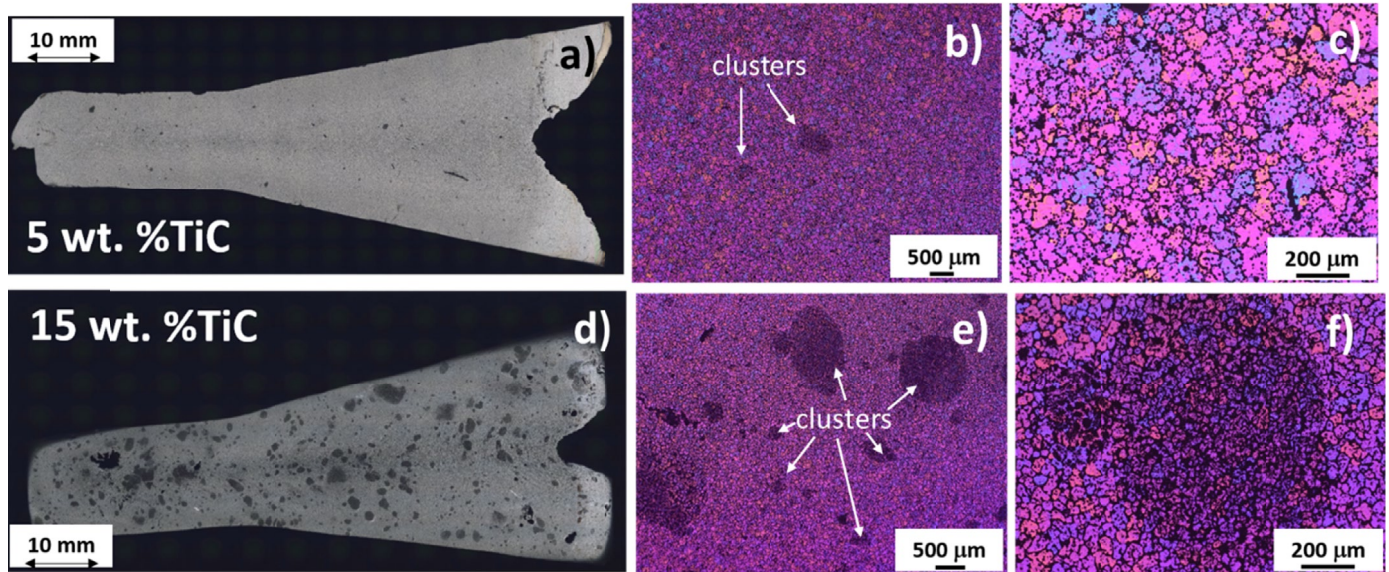


Fig. 1. Set of micrographs of two ingots with 5 wt.% TiC (a,b,c) and 15 wt.%TiC (d,e,f)

individual clusters of particles can be observed. The materials with higher content of TiC reveals a large number of clusters composed of particles with the size of up to 1 mm. This can be related to the phenomenon of engulfment and pushing of particles by solidifying interfaces during casting [28-30] whereas its explanation is beyond the scope of this work. However, it is generally known that with the increase of particles amount, the size/area of the clusters rises, which is also visible in Fig. 1a,d. The images taken at polarised light show a significant difference in the grain size between two investigated ingots. Smaller grain size was found in the composite with higher content of TiC particles, even if they also form different size clusters. The ceramic or intermetallic particles [31] influences on a final grain size of MMC as well as aluminium alloys since they can interact with grain boundaries acting as pinning points [24], retarding or stopping their growth. Thus, the increase of volume fraction with

simultaneous decrease of particles size results in much finer microstructure [19]. The presented results allowed to determine the optimal content of TiC particles, which does not cause clusters formation and confirmed the high impact of volume fraction of particles on the grain refinement. Both microstructural phenomena result in the strengthening of the composites through the load transfer effect and Hall-Petch relationship. In order to examine the grain size homogeneity along the casting cross-section, SEM investigations were carried out in three different parts of the castings containing 5 wt.% of TiC, as presented in Fig. 2.

This ingot was selected for the further studies due to small number of clusters, therefore it can be assumed that the TiC particles are more homogeneously distributed throughout the volume of the casting. The grain refinement of aluminium matrix, caused by TiC formation, is visible in all three areas. Interestingly, there is no big difference of grain size between bottom, middle and

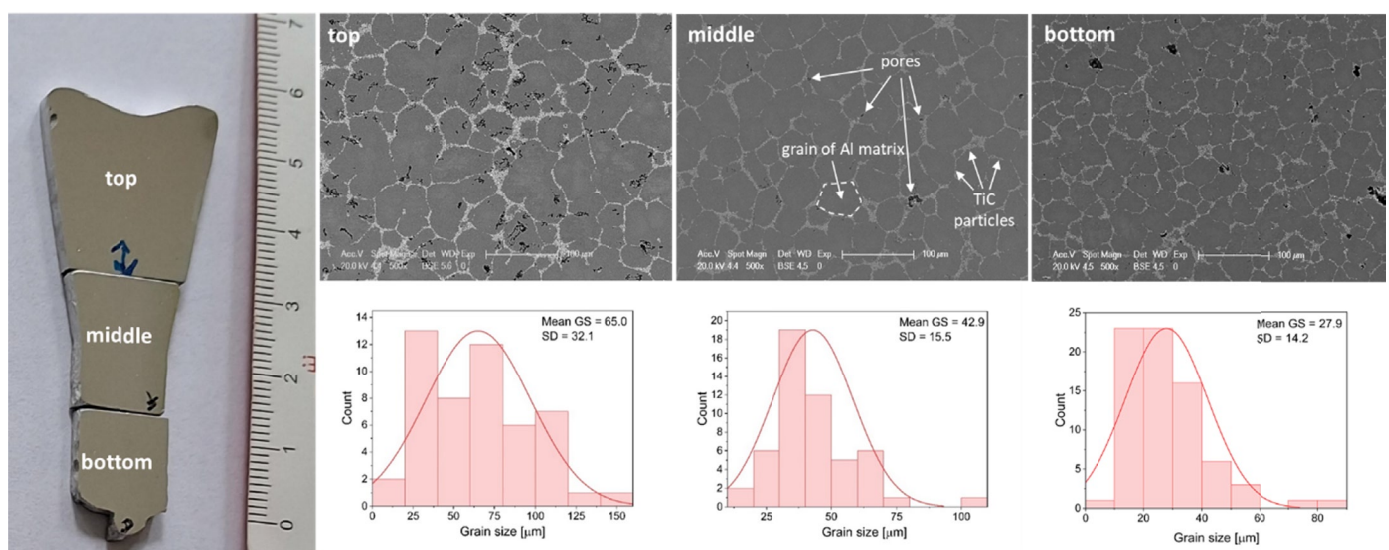


Fig. 2. Cross-section image of the ingot of 5 wt.% TiC/Al composite at low magnification and SEM microstructures recorded in different parts of ingot with corresponding plots of grain size distribution

top part of the presented ingot. This may suggest that dispersion and distribution of TiC particles in molten aluminium, just before the casting, was highly homogeneous. The grain size of aluminium matrix measured by the random secant method is scattered from about 28 μm in the bottom part through 45 μm in middle and finally up to 65 μm in the top. This is accompanied by the temperature gradient during crystallization and also Y type shape of ingot. However, taking into account the standard deviation of grain size measurement as well as their distribution, the most homogenous microstructure appears in the middle part of the ingot. Here, it can be stated that TiC particles formed in-situ in the melted aluminium can be considered as very effective grain refinement of the matrix during crystallization and proper conduct the casting process, especially in terms of mixing of particles in the suspension. The same size and shape of the grains were observed by Wang et al. [32] in TiB₂/Al-Zn-Mg-Cu composite in details has been characterised by SEM-EBSD method including grain boundary description. Other phenomena visible in Fig. 2 on SEM images are the black spots which are representing the porosity. One can see that the highest porosity are located at top part of cast what is typical due to formation of the cavity during crystallization. However, the mean porosity of composite generally does not exceed 1% [24]. Further investigations were focused on microstructural characterization of TiC particles, detection of the additional phases and detailed description of the interface between particles and the matrix. Fig. 3 presents STEM-HAADF image with two square areas indicated by different colours in which qualitative elemental mapping of elements were performed. There are two areas (red and green) differing slightly with contrast in STEM-HAADF image. The elemental maps performed in both areas confirmed the existence of TiC particles but additionally in the case of red one also aluminium oxide can be identified. Moreover, the TiC particles located in the vicinity of oxide are generally much smaller than in other areas. This can suggest that aluminium

oxide can control growth of TiC particles during crystallization. Finally, fine Fe-Al particles also can be identified in the microstructure of composite, commonly present in aluminium alloys as impurities. The crystal structure of aluminium matrix and aluminium oxide identified by the elemental mapping were confirmed by the electron diffractions.

Fig. 4 shows an images of the microstructure obtained in bright-field TEM (TEM-BF) and corresponding selected area electron diffraction (SAED) patterns of blocky like aluminium oxide particle embedded in the aluminium matrix. SAED patterns taken from the area of oxide particles can be well indexed in accordance to the [0001] zone axis of hexagonal Al₂O₃ oxide. The Al₂O₃ particles are embedded in Al matrix, which was confirmed by the SAED patterns. However, the TEM-BF images of microstructure recorded with two different tilts shows also a plenty of TiC particles (indicated by arrows) located both in the Al matrix as well as in the Al₂O₃ blocky particle. Moreover, TiC particles located in the oxides are much smaller than those existed in the matrix while their size is below 100 nm. This can suggests that aluminium oxides can be an effective factor inhibiting the growth of TiC particles formed in-situ during the casting process. Finally, this can influence on the mechanical properties of composites assuming the Orowan strengthening. One of most important issues affecting the mechanical properties of composites is strong interface between the matrix and reinforced particles, including also lattice mismatch of these two components.

Since the Al matrix and TiC particles have the same *fcc* crystal structure but differs with lattice parameters while the theoretical lattice mismatch is of about 6.8% (Fig. 5). The faceted TiC particle of size of about 500 nm surrounded by the Al matrix is presented in Fig. 5. The SAED patterns for both the particle and the matrix have the same [011] zone axis, nevertheless the Al matrix is in Bragg condition (dark phase in BF-TEM image) whereas TiC is slightly off-axis (no symmetrical diffraction and

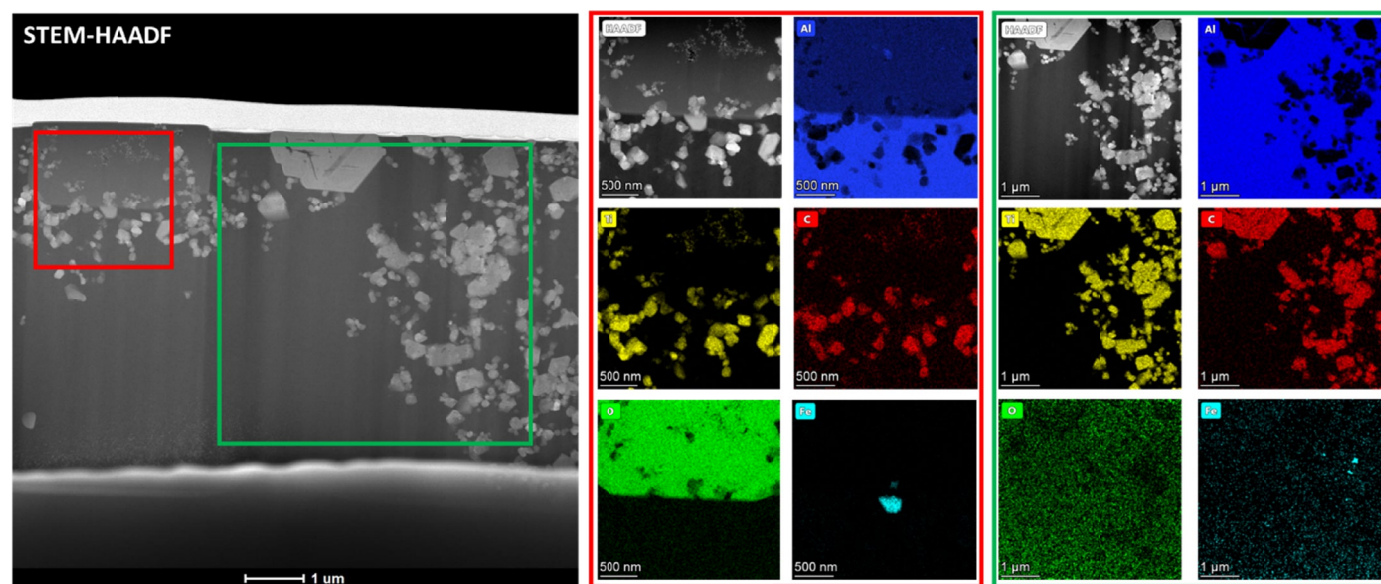


Fig. 3. STEM-HAADF image with two square areas indicated by different colours in which qualitative elemental mapping of elements were performed

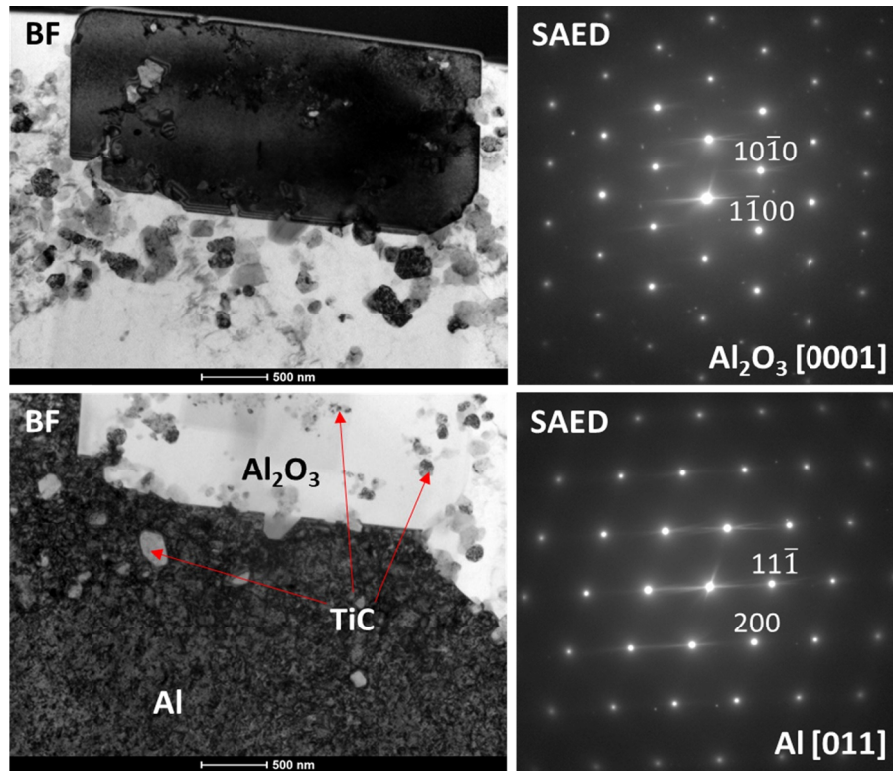


Fig. 4. BF-TEM images and SAED patterns of blocky like aluminium oxide particle embedded in aluminium matrix

contrast in BF-TEM image is grey) due to lattice mismatch. Based on electron diffraction patterns, it can be assumed that individual crystallographic planes form interfaces between the particle and the matrix. Moreover, considering that the TiC particle is a typical faceted crystal, the specific crystallographic planes correspond exactly to the walls of the TiC crystal. One can see that $\{200\}$ and $\{111\}$ type planes both of Al and TiC are almost parallel and forms strong interface visible in HRTEM

image. There is a few nanometres band between Al and TiC enriched with misfit dislocations formed due to lattice mismatch. However, interface composed of $\{111\}$ type planes both of TiC and Al is declined of about 15° of each other.

This can introduce more structural faults but this issue is beyond of this article and will be developed in the further investigations. Nevertheless, the influence of the lattice mismatch on the strengthening of the investigated composite is already visible

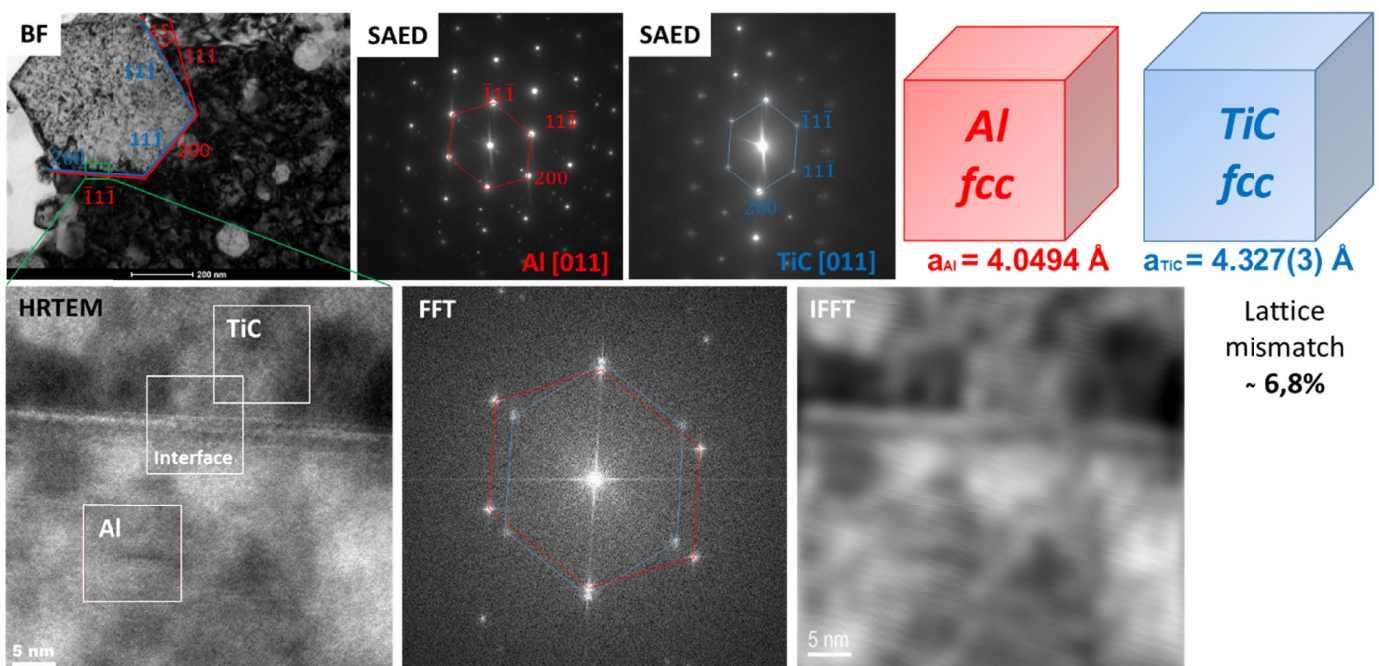


Fig. 5. BF-TEM image and corresponding SAED patterns of Al and TiC and HRTEM showing the interface between Al matrix and TiC particle

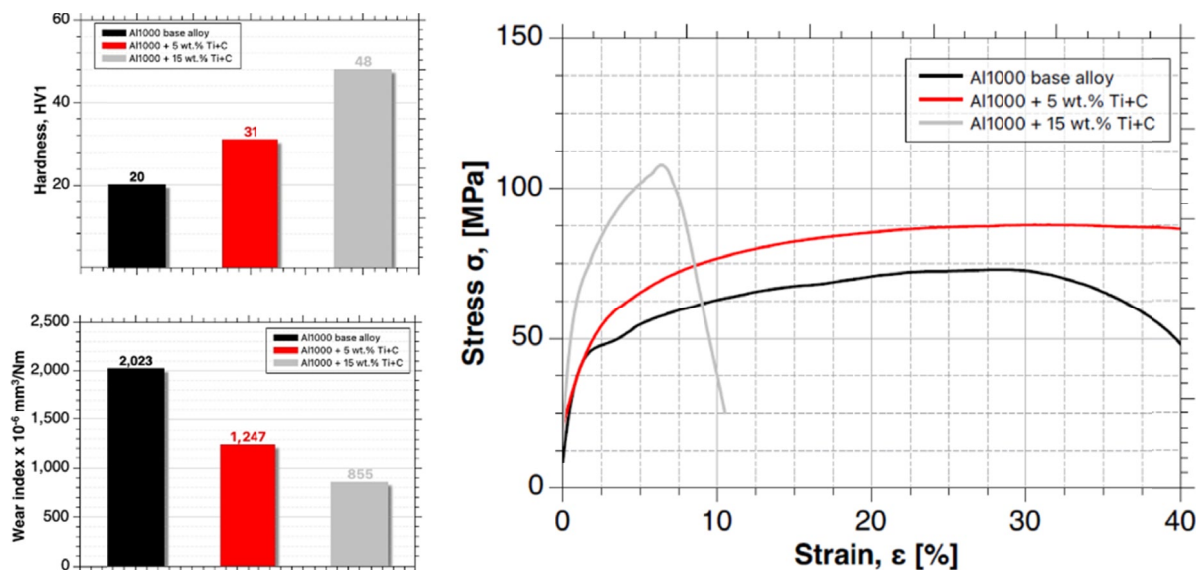


Fig. 6. Set of mechanical properties and wear index of Al1000 alloy and composites with 5 and 15 wt.% of TC particles

at this point. In the above considerations, we have shown that multiscale microstructure analysis helps to describe some of the effects related to MMC strengthening. They influence on both the overall strength and hardness as well as functional properties such as abrasive wear resistance. As can be seen in Fig. 6, both hardness, tensile strength and abrasive wear resistance were improved with the addition and increase of the volume fraction of TiC particles. Moreover, addition of 15 wt.% of TiC to the aluminium is more beneficial than 5 wt.% even if the clustering process of TiC particles was intensified as was shown in Fig. 1.

According to publication [33] for composite with the reinforcement particle of size of about 150 nm the most important contributions for strengthening is CTE mismatch associated with formation of geometrically necessary dislocations (GNDs) and then Hall Pech and Orowan effects connected with grain refinement and pinning of dislocation movement under external load. These effects also plays the major role in strengthening of composite in our case. The bigger number of TiC particles causes formation of more amount of GNDs and bigger grain refinement. Moreover, increase of number of TiC particles simultaneously increases hardness and wear resistance of composite since TiC particles has higher hardness then the Al matrix. This is in good agreement with result presented in Fig. 6.

4. Conclusions

Using multiscale microstructural analysis of in-situ cast AMCs starting from light microscopy through SEM and ending with TEM we have shown which strengthening effect play the main role. The following conclusions can be drawn from the analysis of the obtained results:

1. The refinement of the grain size of composite matrix (Hall-Patch relationship) occurs with the increase of TiC particles amount. The clustering effect appears in the composite with

15 wt.% TiC and this can be related to the phenomenon of engulfment and pushing of particles by solidifying interfaces during casting.

2. TiC particles in-situ formed in the melted aluminium can be considered as very effective grain refinement of the matrix during crystallization and proper conduct of the casting process, especially in terms of mixing of suspension of particles. This can lead to high quality composite ingot characterized by homogeneous grains in the entire volume of the ingot.
3. Lattice mismatch of Al and TiC due to their different lattice parameters introduces additional structural faults like misfit dislocations in the few nanometre band located at the interface. The formation of oxide particles as a consequence of oxidation of aluminium during casting can affect the TiC particles size.
4. Addition of 15 wt.% of TiC particles to the aluminium matrix is more beneficial for mechanical properties and wear resistance than 5 wt.% even the intensified clustering process of TiC particles.

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