UDK 621.74:620.18:669.13
Professional article/Strokovni članek

ISSN 1580-2949
MTAEC9, 50(3)451(2016)

MICROSCOPIC CHARACTERIZATION AND PARTICLE DISTRIBUTION IN A CAST STEEL MATRIX COMPOSITE

MIKROSKOPSKA KARAKTERIZACIJA IN RAZPOREDITEV DELCEV V KOMPOZITU Z MATRICO LITEGA JEKLA

Ana Kračun[1,2], Matjaž Torkar[1], Jaka Burja[1], Bojan Podgornik[1]

[1]Institute of Metals and Technology, Lepi pot 11, 1000 Ljubljana, Slovenia
[2]International postgraduate school Jožef Stefan, 1000 Ljubljana, Slovenia
ana.kracun@imt.si

Prejem rokopisa – received: 2015-10-01; sprejem za objavo – accepted for publication: 2015-12-24

doi:10.17222/mit.2015.310

The purpose of this investigation was to identify the distribution of ultrafine particles in a steel matrix introduced through a conventional melting and casting method, and above all to determine the methodology and analysing techniques suitable for the analysis and identification of ultrafine particles incorporated into the steel matrix. In the frame of this work, steels dispersed with Al_2O_3 ultrafine particles were produced by a conventional casting method and their microstructure investigated with light microscopy (LM), scanning electron microscopy (SEM) and auger electron spectroscopy (AES). Microstructural analyses show that the distribution of the Al_2O_3 ultrafine particles is non-uniform and has a high degree of agglomeration. Furthermore, for a detailed analysis of the nanoparticles a specific preparation and characterization using advanced microscopic techniques is required.

Keywords: particle distribution, microscopic characterization, steel matrix

Namen raziskave je bil ugotoviti porazdelitev ultrafinih delcev v jekleni matrici, ki je bila proizvedena s konvencionalnim postopkom litja, predvsem pa določiti metodologijo in analizne tehnike, primerne za analizo in identifikacijo ultrafinih delcev, ki so bili vključeni v jekleno matrico. Delci Al_2O_3 so bili dodani med procesom konvencionalnega litja in so bili analizirani s pomočjo različnih analiznih tehnik, in sicer: z uporabo optičnega mikroskopa (LM), vrstičnega elektronskega mikroskopa (SEM) in spekroskopije Augerjevih elektronov (AES). Analiza mikrostrukture je pokazala neenakomerno porazdelitev in aglomeracijo Al_2O_3 delcev. Za podrobno analizo je potrebna karakterizacija mikrostrukture s pomočjo naprednih mikroskopskih tehnik.

Ključne besede: porazdelitev delcev, mikroskopska karakterizacija, jeklena matrica

1 INTRODUCTION

The insertion of ceramic reinforcements into metal matrices to produce composite materials with improved properties has been a subject of intensive research during the past three decades.[1–3] Ceramic particulates such as borides, carbides, oxides and nitrides are added to metal matrix composites (MMCs) to improve their elastic modulus, wear resistance, creep and strength.[4–5]

The ductility of MMCs, however, deteriorates at high ceramic particle concentrations[5]. The metal matrix, the so-called metal-matrix nano-composite (MMnCs) is strengthened by nano-sized ceramic particles.[6] These nanoparticle reinforcements can significantly increase the mechanical strength of the metal matrix, as they promote particle hardening more effectively than micro particles. Moreover, MMnCs improve the performance significantly at elevated temperatures, because the ceramic nanoparticles can maintain their properties at high temperatures.[6]

Steel matrix composites commonly have a combination of hard ceramic (e.g., TiC, TiB_2, WC and Al_2O_3) reinforcements and a ductile metallic matrix, which makes them promising candidates for high-strength and wear-resistance applications. There are several methods for fabricating particulate-reinforced steel matrix composites, such as powder metallurgy, conventional melting and casting, reactive sintering and self-propagating high-temperature synthesis (SHS). The casting process is simple and more economical than the other available routes for integrating nanoparticles into the microstructure of steel. However, it is extremely difficult to obtain a uniform dispersion of ceramic nanoparticles in liquid metals due to the poor wettability and the difference in the specific gravity between the ceramic particles and the metal matrix.[7]

The microstructure of metals is generally characterized by advanced microscopic techniques (e.g., LM, SEM and TEM) which probe and map the surface and sub-surface structure of a material. These techniques can use photons, electrons, ions or physical cantilever probes to gather data about a sample's structure on a wide range of length scales.[8] Auger electron spectroscopy (AES) also provides quantitative elemental information from the surfaces of solid materials.[9]

The current work aims at contributing to the knowledge and understanding of the conventional casting route for ultrafine particle inoculation in a steel matrix. This

production route seems to show potential and offers more cost efficiency in achieving the dispersion of second-phase ultrafine particles compared to the powder and metallurgical techniques used until now. The aim of the work was therefore to study the influence of Al_2O_3 ultrafine particles on the microstructure of a steel matrix using a conventional casting method. The additional aim is to determine the methodology and analysing techniques suitable for analysing and necessary to identify the ultrafine particles incorporated in the steel matrix.

2 EXPERIMENTAL WORK

2.1 Material

Austenitic stainless steel was used for the work, mainly due to the distinctive two-phase microstructure of austenite and ferrite. The chemical composition of this alloy is given in **Table 1**. These are the most used group of stainless steels. They are paramagnetic, have a face-centred cubic lattice and excel with a good combination of hot and cold workability, mechanical properties and corrosion resistance.

Table 1: Chemical composition of austenitic stainless steel in mass fractions, (*w*/%)
Tabela 1: Kemijska sestava avstenitnega nerjavnega jekla v masnih deležih, (*w*/%)

Elements	*w*/%
C	0.02
Si	0.33
Mn	1.24
Cr	17.4
Ni	10.1
Cu	0.36
Mo	1.29
V	0.08

As the reinforcement particles, commercial ultrafine Al_2O_3 powder with a mean particle size of 500 nm was used, as shown in **Figure 1**. The Al_2O_3 ultrafine particles were selected due to their high chemical stability to Fe and high specific gravity. In particular, it was reported that the wetting angle θ between Al_2O_3 and molten iron alloy is less than 50°, even at high temperatures and in many different types of atmospheres.[10]

2.2 Specimens preparation

A weighed quantity (10 kg) of the austenitic stainless steel was melted in an induction furnace. In the first experiment 20 g of the ultrafine Al_2O_3 particles were wrapped in an Al foil and put into the ingot and the molten metal was poured over it into the same ingot. In the second experiment a mixture of 24 g of Al_2O_3 and 2.4 g of dry glue was prepared. The mixture was then filled in the steel tube and flooded with paraffin. The tube was inserted into the molten metal and when melted, the molten metal was poured into the ingot.

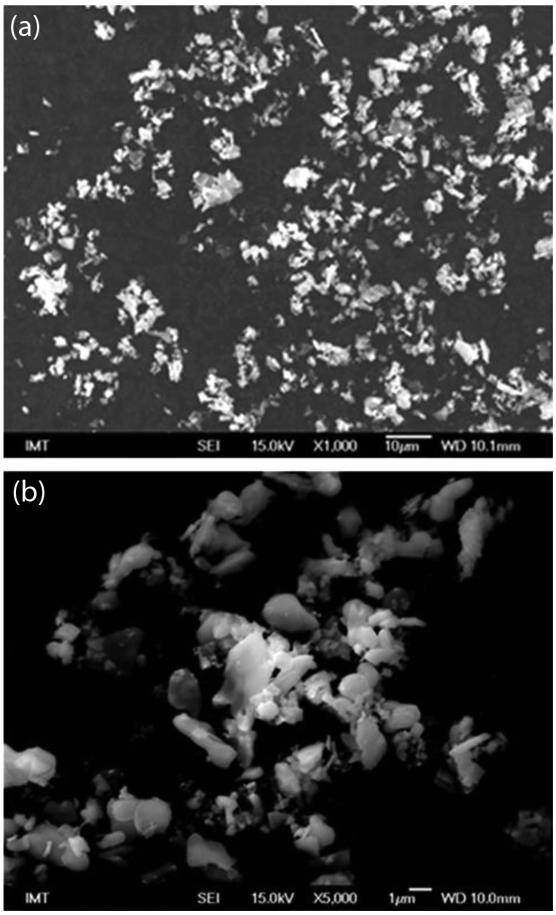

Figure 1: SEM image of Al_2O_3 ultrafine particles at various magnifications
Slika 1: SEM-posnetek ultrafinih delcev Al_2O_3 pri različnih povečavah

2.3 Characterization

The microstructural changes and the dispersion of the ceramic particles in the steel matrix were observed and analysed using light microscopy (LM), scanning electron microscopy (SEM) and auger electron spectroscopy (AES). Samples for the microstructure analysis were taken from the bottom, middle and top portions of the cast piece. Metallographic samples were prepared by grinding, polishing, followed by chemical etching and analysed to reveal the particle distribution. Samples for Auger electron spectroscopy were prepared by grinding and polishing the surface. These samples were attached to the bracket, placed in an experimental container-air-lock, pumped to UHV and transferred into an analytical container. The surface of the sample was ion etched and analysed to determine the elemental composition in the surface region of the sample.

3 RESULTS AND DISCUSSION

Figure 2 shows a LM micrograph of the microstructure of pure austenitic stainless steel with a distinctive

two-phase microstructure of austenite and δ-ferrites. A LM micrograph of the microstructure and ultrafine particles' distribution of the sample produced by the casting process of the austenitic stainless steel poured over the Al_2O_3 ultrafine particles is shown in **Figure 3**.

As shown in **Figure 3**, the microstructure of the austenitic stainless steel is modified after the addition of Al_2O_3 ultrafine particles, being incorporated into the metal matrix. However, the distribution of Al_2O_3 particles is non-homogeneous and concentrated in a certain area.

In **Figure 4** the particle distribution of the sample taken from the second experiment, where the steel tube filled with Al_2O_3 particles was inserted into the melt is shown. As in the case of the first experiment, with the molten steel being poured over the Al_2O_3 ultrafine particles, the distribution of the particles is non-uniform and has a high degree of agglomeration (**Figure 4**). However, the degree of particles is lower when inserting the particles-filled steel tube into the molten metal.

From the SEM elemental analysis, shown in **Figure 5**, it was confirmed that the bright, small, spot-like features represent the Al_2O_3 ultrafine particles that are non-uniformly distributed in the steel matrix.

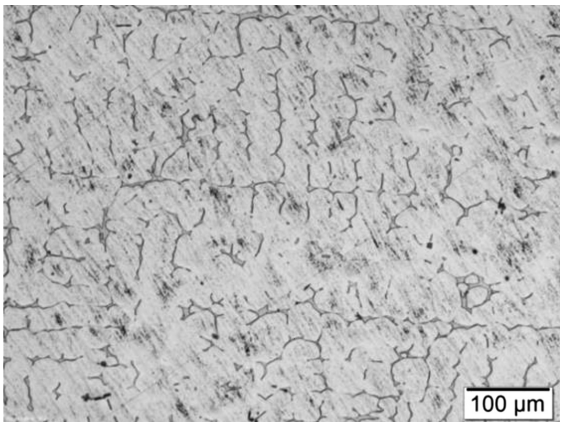

Figure 2: Cast microstructure of austenitic stainless steel with 6 % of δ-ferrite

Slika 2: Lita mikrostruktura avstenitnega nerjavnega jekla s 6 % δ-ferita

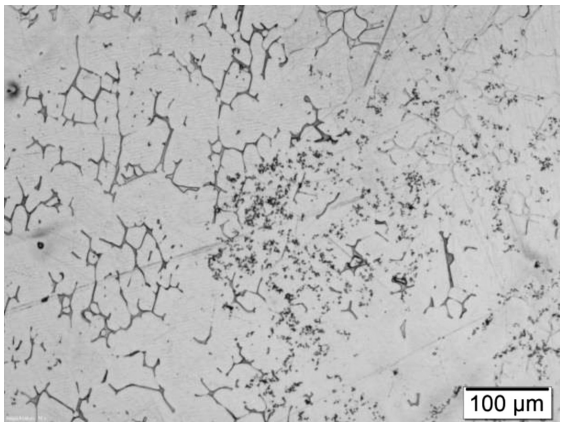

Figure 3: Cast microstructure of austenitic stainless steel with 6 % of δ-ferrite and Al_2O_3 ultrafine particles

Slika 3: Lita mikrostruktura avstenitnega nerjavnega jekla s 6 % δ-ferita in Al_2O_3 ultrafinimi delci

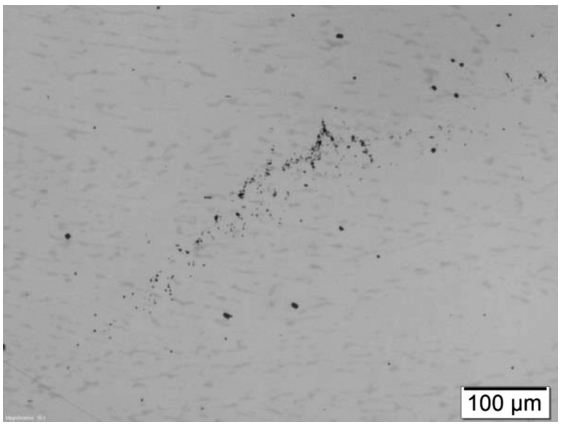

Figure 4: Cast microstructure of austenitic stainless steel with Al_2O_3 ultrafine particles inserted into the melt (experiment 2)

Slika 4: Lita mikrostruktura avstenitnega nerjavnega jekla z Al_2O_3 ultrafinimi delci, vstavljenimi v talino (preizkus 2)

In **Figure 6** the AES spectrum of the Al_2O_3 ultrafine particles in the cast microstructure of austenitic stainless steel is shown. The spectra of particles (P1 and P2) showing only O and Al peaks confirm the successful introduction of Al_2O_3 ultrafine particles into the steel matrix (P3) without any intermetallic reaction taking place.

4 CONCLUSIONS

Steel matrix composites with non-uniformly dispersed Al_2O_3 ultrafine particles were produced by a conventional melting and casting method. The purpose of this investigation was to determine the methodology and analysing techniques suitable for the analysis and identi-

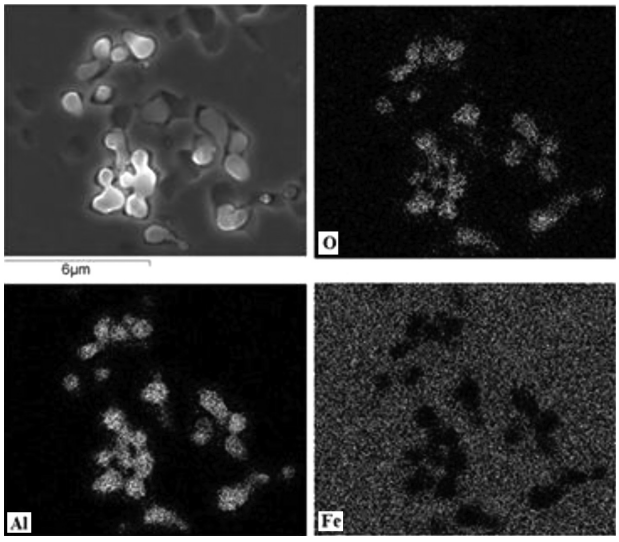

Figure 5: SEM elemental analysis of Al_2O_3 ultrafine particles in the cast microstructure of austenitic stainless steel

Slika 5: SEM-posnetek elementne analize Al_2O_3 ultrafinih delcev v liti mikrostrukturi avstenitnega nerjavnega jekla

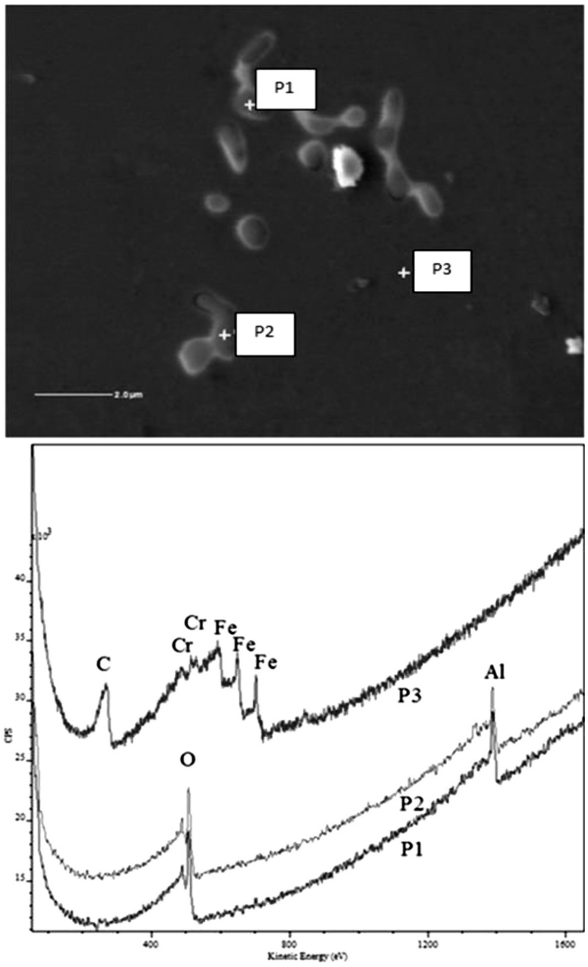

Figure 6: AES spectrum of the Al_2O_3 ultrafine particles in the cast microstructure of austenitic stainless steel

Slika 6: AES-spekter analize Al_2O_3 ultrafinih delcev v liti mikrostrukturi avstenitnega nerjavnega jekla

fication of ultrafine particles incorporated in the steel matrix.

The microstructural changes and the dispersion of the Al_2O_3 ultrafine particles in the steel matrix were observed and analysed by light microscopy (LM), scanning electron microscopy (SEM) and auger electron spectroscopy (AES). This work clearly shows that for a proper analysis and identification of the successful nano-particles' incorporation, different analysing techniques need to be used and combined.

Based on the experimental results the dispersion of the Al_2O_3 ultrafine particles in the steel matrix is non-homogeneous and concentrated in certain areas.

In order to be able to obtain a homogeneous distribution of reinforcements in the metal matrices the following factors need to be understood and taken into consideration for future work:

- particle density, size, shape and volume fraction will influence the reinforcement settling rate,
- surface properties of the particles will affect the wetting with molten metal,
- rheological behaviour is influenced by the reaction of the particles with the melt and each other,
- in general, the reinforcement particles occupy inter-dendritic or between secondary dendrite arm spacings, while the particle distribution is also metal-matrix dependent.

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