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METALLURGY

DOI: 10.2478/amm-2013-0069

Volume 58

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COMPOSITE ZONES OBTAINED BY IN SITU SYNTHESIS IN STEEL CASTINGS

STREFY KOMPOZYTOWE OTRZYMYWANE IN SITU W ODLEWACH STALIWNYCH

Application of in-situ technique allows for fabrication of composite zone in the casting with a matrix of cast steel with low carbon content. The reinforcing phase in the composite zone is titanium carbide, produced by the synthesis of substrates introduced into the mould in the form of pressed compacts. Metallographic studies were performed in order to determine of homogeneity of composite zones and characteristic features existed in transition area between the composite zone and core of the casting. The transition areas of composites zone were blurred and there were no discontinuities caused by poor bonding between the composite zone and the core of the casting. To confirm the correct run of the TiC synthesis, phase analysis was performed of the base alloy as well as composite zone. The results of this examination indicated that there were two phases in composite zone, i.e. α Fe and TiC and only α Fe was observed in base alloy. Changes of mechanical properties in the composite zone were examined, measuring its hardness HV. The average hardness values of the base alloy and composite zone were 175±4 and 696±201 HV, respectively.

Keywords: composite zone, in situ, TiC, MMC, cast steel, microstructure

W odlewie z ferrytycznego staliwa o niskiej zawartości węgla, otrzymano strefy kompozytowe. Fazą wzmacniającą strefy kompozytowej był węglik tytanu TiC, uzyskany w wyniku syntezy in situ substratów, wprowadzonych do formy w postaci wyprasek. Przeprowadzono badania metalograficzne stref kompozytowych w celu określenia jednorodności otrzymanej strefy kompozytowej oraz charakteru obszaru przejściowego: strefa kompozytowa – rdzeń odlewu. Obszar przejściowy, pomiędzy strefą kompozytową, a rdzeniem odlewu, charakteryzował się gradientowym rozkładem TiC oraz brakiem nieciągłości. W celu potwierdzenia prawidłowego przebiegu procesu syntezy TiC, przeprowadzono analizę fazową stopu bazowego oraz otrzymanej strefy kompozytowej. Potwierdzono, że w obrębie strefy kompozytowej występują dwie fazy tj. α Fe i TiC, a stop bazowy zawiera wyłącznie α Fe.

Analizowano zmiany mechaniczne w obrębie otrzymanej strefy kompozytowej, badając jej twardość HV. średnia wartość twardości stopu bazowego oraz strefy kompozytowej wynosiła odpowiednio 175±4 i 696±201 HV.

1. Introduction

Abrasion wear is one of the major processes causing damage to the structural components of industry equipment. To eliminate this adverse effect, parts of machines and equipment are produced from alloys, cermets, stellites and sintered materials characterised by high abrasion resistance. The selection of material for a given component should allow for conditions under which this component will be operating in the future. Often, it is subjected to large dynamic loads, and then it is the toughness of the material that will decide on its further applicability. A large group of abrasion wear-resistant materials includes cast iron alloys. Their main advantage is a relatively low cost of production and a relatively easy technology to make parts with complex shapes and large overall dimensions. Another important factor is also the possibility to design in a wide range of values, their mechanical and performance properties, mainly through an impact exerted on

the microstructure. In the family of iron alloys, the materials most commonly used for the abrasion resistant components are selected grades of the cast iron and cast steel.

Abrasion-resistant cast steels are mainly used in mining and cement industries, in processing of metal ores and minerals, and in other sectors of production. The basic use of steel castings in these areas is for the components of crushers, mills and excavators like, among others, hammers, plates, cones, rings and balls, teeth, buckets, track links, etc. (Fig. 1).

For the cast machine parts exposed to wear under high dynamic loads, the most commonly used material is Hadfield cast steel, mainly due to its high toughness and impact resistance. This high-manganese cast steel (1.2% C and 12% Mn), which was invented in 1882 by Sir R.A. Hadfield [1]. Currently, numerous modifications of this cast steel are known and available, mainly by providing a higher manganese content of up to $20 \div 30\%$ [2], and the addition of carbide-forming elements, such as chromium, molybdenum and vanadium [3,4]. On one

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hand, these additions increase the wear resistance while, on the other, they also increase the cost of the casting production due to the high price of alloying elements and the need to subject this cast steel to a high-temperature heat treatment. Among the available grades of casting alloys, there are no compositions that could replace the Hadfield cast steel and provide so excellent wear resistance combined with equally good toughness. Under the conditions of impact loads, only plastic alloys can offer the required properties and ensure the reliable and safe operation of machinery and equipment.



Fig. 1. Examples of castings where the composite zones are useful; hammer (a), excavator tooth (b)

However, when the main wear process is friction, Hadfield cast steel suffers the destruction very rapidly, especially in abrasive environments.

A solution to this problem may be two-layer castings [5-7], inserts made from sintered carbides, remelted surface layers [8] and, finally, the MMC type composites, i.e. Metal Matrix Composites [9]. In the MMC family, two main development trends are observed, i.e. composites fabricated by the ex situ methods [10-13] and in situ methods [14-16]. One of the techniques of producing in situ composites is by SHS (Self-Propagating High-Temperature Synthesis) and SHSB process (Self-Propagating High-Temperature Synthesis in Bath) [17-20]. In this method, the reinforcing phase is formed by a spontaneous, exothermic chemical reaction in the metal bath. The composite manufacture is a single-step process. This method also enables, in addition to the fabrication of the reinforcing phase within the total volume of the alloy, local reinforcement in selected areas of the casting [23]. The process is carried out by placing substrates of the carbide synthesis in the mould cavity and pouring the mould with liquid alloy next. As a consequence of the reaction of synthesis proceeding in the liquid alloy, carbides are formed in selected parts of the casting.

In this study, a method for the in situ fabrication of composite zones in castings based on ferritic steel was described. The influence of the process of synthesis on changes of microstructure within the composite zone and in the base alloy was analysed. Vickers hardness of the obtained composite zone was also measured.

2. Experimental

The mixture of substrates necessary for the synthesis of titanium carbide was prepared in an atomic ratio of 1:1, according to the reaction in equation (1).

$$Ti + C = TiC \tag{1}$$

For this purpose, the commercial products such as powders of titanium (99.98 %, 44 μ m) and graphite (99.99 %, 44 μ m) were used. The pure powders were mixed without the access of air for 24 hours, and then five compacts with dimensions of 10×12×55 mm, weighing 0.003 kg each were consolidated at a pressure of 500 MPa.

The base alloy selected for investigations was unalloyed, low-carbon, ferritic cast steel. The selection of this particular material was dictated by the need to obtain a plastic core in the casting ensuring high fracture toughness. The chemical composition of the base alloy is given in Table 1.

TABLE 1

Chemical composition of the base cast steel

Chemical composition, [wt. %]										
C	Si	Mn	Р	S	Cr	Ni	Fe			
0.054	0.077	0.094	0.001	0.007	0.28	0.05	balance			

Cast steel was melted in a medium frequency induction furnace with a 20 kg capacity crucible. The charge consisted of an unalloyed steel scrap, grade S235, and Armco was iron. After melting, the steel was deoxidised with 0.02 kg of aluminium and 0.05 kg of FeTiAl. Then it was superheated to a temperature of 1600°C and tapped. During tapping, the steel was deoxidised with aluminium in an amount of 0.01 kg and with FeCa30Si in an amount of 0.02 kg.

Before pouring of mould, the produced compacts with substrates for the TiC synthesis were installed in mould. The schematic diagram of mould with compacts placed in the mould cavity is shown in Figure 2.



Fig. 2. Schematic diagram of foundry mould with substrates necessary for the TiC reaction of synthesis

The alloy was next poured into a test mould made of a furan-bonded silica sand mixture. The base alloy casting temperature was 1625° C.

The final casting was cut, ground and polished to perform metallographic investigations and mechanical tests.

The chemical composition of the alloy was examined by optical spectrometry, using SpectroMAXx emission spark-induced spectrometer made by Spectro. The crystallographic structure of the base alloy and of the composite zone was determined with Kristalloflex 4H X-ray type diffractometer made by Siemens using CuK α radiation (0.15418 nm). The research was carried out at a voltage of 30 kV and a current of 20 mA. The microstructure investigations both the base alloy and composite zones were done by SEM using an SU-70 Hitachi scanning electron microscope. Backscattered electrons (BSE) operation mode was also applied. Vickers hardness measurements were taken on the obtained materials using an HPO 250 hardness tester, a load of 294.2 N and the load application time of 15 seconds.

3. Results

Figure 3 a and b shows the BSE images of the microstructure produced in a transition area between the composite zone and the core of casting. In Figure 3 a and b, the dark precipitates of TiC obtained as a result of the in situ synthesis in liquid alloy are distinctly visible. The carbides are distributed in the light-colour matrix. The interface between composite zone and casting core is clearly visible, and there is no cracks and discontinuities.

Figure 3 c and d shows the BSE images of the microstructure of composite zone. Within this zone (Fig. 3c), clusters of carbides, forming conglomerates and coagulated shapes, are observed. Between these areas, numerous non-faceted, randomly spaced carbides of oval and dendritic shapes appear in the matrix. The resulting composite zone is characterised by heterogeneous distribution and large number of the coagulated titanium carbides. Carbides shown in Figure 3 d form irregular, closed areas, inside which the alloy matrix is visible and randomly distributed TiC dendrites.



Fig. 3. BSE images of microstructure in the transition area between the composite zone and casting core (a-b) and composite zone produced in casting (c-d)

Figure 4 shows the BSE image of the microstructure of the composite zone with marked areas of the energy dispersive X-ray (EDX) microanalysis. The results of the EDX microanalysis are presented in Table 2. In areas 3 and 4 of the composite zone, carbon and titanium were mainly identified. This result may indicate the presence of phases from the Ti-C system, such as e.g. TiC. In areas 1 and 2, iron is mainly present. The results of the microanalysis of the chemical composition are confirmed by the phase composition of the examined material (Fig. 5).



Fig. 4. BSE image of the composite zone with marked areas of the EDX microanalysis

TABLE 2
Results of the EDX microanalysis of composite zones performed in
marked areas in Figure 4

	Chemical composition, [wt. %]						
Area Zone No.	C	Al	Ti	Cr	Fe		
1	-	0.2	0.5	0.2	balance		
2	-	0.3	0.3	0.2	balance		
3	13.7	0.1	85.7	-	0.3		
4	13.7	0.1	85.8	-	0.4		



Fig. 5. Results of phase analysis of the base alloy and composite zone area (a) and 110 and 211 reflections of α Fe phase presented in the narrow 2 Theta range (b) and (c) respectively

Figure 5a shows the results of X-ray diffraction on crystallographic planes of structures obtained in the casting core and composite zone produced in this casting. The Bragg reflections 110, 200, 211 and 220, visible on diffraction patterns of the casting core and composite zone, originate from the structure of α Fe (ferrite) classified in the space group 229 with an abbreviated symbol of Im-3m. Other reflections visible on the diffraction pattern of the composite zone originate from the structure of titanium carbide with the space group number 225 and symbol Fm-3m. The reflections visible in Figure 5 b and c, derived from the (110) and (211) family of planes in the structure of α Fe, show no displacements relative to each other. This result indicates absence of changes in the dimensions of

the α Fe crystal cells occurring within the composite zone. Figure 6 illustrates the results of Vickers hardness measurements taken on the base alloy and in the composite zone. The average hardness values of the base alloy and composite zone are 175 ± 4 and 696 ± 201 HV, respectively. In the composite zone produced in casting, an average four time increase of hardness in relation to the base alloy was obtained. Additionally, in areas with the highest concentration of the reinforcing phase, this value was even six times higher. Due to the previously described heterogeneous distribution of carbides in the composite zone, the scatter of the hardness results is very large.



Fig. 6. Results of HV hardness measurements obtained for the casting core and composite zone

4. Discussion

The process of the in situ synthesis of titanium carbide was carried out directly in the liquid melt. Substrates for the reaction of the carbide synthesis were in the form of pressed compacts placed directly in the mould cavity and then poured with base alloy. After the casting solidification, in the place where substrates had been placed, composite zones were obtained with the dimensions corresponding to the dimensions of compacts introduced into the mould. The X-ray diffraction showed two basic structures present in these zones, i.e. α Fe and TiC (Fig. 5). Phase analysis showed no changes in the lattice parameters of both mentioned phases. This indicates that substrates did not dissolve in base alloy, especially as regards titanium carbide. This carbide occurs in a wide range of the chemical composition in a Ti-C phase equilibrium diagram.

The microstructure investigations revealed high heterogeneity level of the composite zone (Fig. 3). Within this area, numerous coagulated carbides, forming compact agglomerates of large dimensions, were present. Between them there were areas with TiC dendrites randomly distributed in the matrix (Fig. 3c). The coagulation process of titanium carbide could be caused by the base alloy solidification conditions and the related carbide growth kinetics (Fig. 3d). This phenomenon was discussed in [23], where a relationship was derived between the growth of carbides in function of time during which they rest in liquid alloy.

Hardness measurements taken in the obtained composite zones showed an average fourfold increase of hardness relative to the base alloy with values amounting to ± 700 HV and ± 175 HV, respectively (Fig. 6). In extreme cases, a sixfold increase relative to the base alloy was also obtained, and then the value of hardness amounted to as much as 1000 HV. This result can explain the high heterogeneity of microstructure in the composite zone, where the surface content of carbides varies considerably.

Hard composite zones generated in castings made from the alloy characterised by high toughness and impact strength allowed implementation of the two main objectives of the study. The first objective was to produce the surface with high hardness, and this goal was obtained by producing a composite zone in the casting. The second objective concerned toughness and was obtained by producing a plastic core in the same casting.

Thus, the resulting material meets the requirements established by the authors of the study. However, to get uniform hardness levels in the zone, the phenomenon of the TiC crystal growth and coagulation needs to be mitigated.

5. Conclusions

The SEM observation reveals, that TiC particles has nearly oval and dendritic in shapes. There is no other particles such oxides and nitrides between TiC and metal matrix grain boundary. No decohezion of TiC particles from the matrix can be observed. The hardness of composite layer is three or even four times higher than hardness of matrix. The X-ray diffraction on crystallographic planes of structures indicates that there is no changes in the dimensions of α Fe crystal cell in the composite zone. It reveal that substrates (Ti and C) does not make solid solution with iron.

Acknowledgements

The research formed part of the study No. N N507 311040 financed by the Polish Ministry of Science and Higher Education.

REFERENCES

- [1] G. Tweedale, Notes & Rec. of the Royal Society, London (1985), DOI: 10.1098/rsnr.1985.0004.
- [2] A.K. Srivastava, K. Das, Materials Science and Engineering A **516**, 1 (2009).
- [3] J. Krawiarz, L. Magalas, Przegląd Odlewnictwa **10**, 666 (2005).
- [4] B. Kalandyk, R. Zapała, Archives of Foundry Engineering **9(4)**, 91 (2009).
- [5] M. Cholewa, T. Wróbel, S. Tenerowicz, Journal of Achievements Materials and Manufacturing Engineering 43(1), 389 (2010).

- [6] J. Suchoń, Archives of Foundry Engineering 10, 214 (2003).
- [7] J. Suchoń, Archives of Foundry Engineering **22**, 483 (2006).
- [8] M. Rozmus-Górnikowska, J. Kusiński, M. Blicharski, Archives of Metallurgy and Materials 56(3), 717 (2011).
- [9] D.B. Miracle, Composites Science and Technology 65, 2526 (2005).
- [10] R. Asthana, Journal of the Material Science 33, 1679 (1998).
- [11] J. M y a l s k i, J. Ś l e z i o n a, Journal of Materials Processing Technology 175, 291 (2006).
- [12] A. Zyska, J. Braszczyński, Z. Konopka, Kompozyty (Composites) 1, 114 (2001).
- [13] J. Lelito, P. Żak, J.Sz. Suchy, Archives of Metallurgy and Materials 54(2), 347 (2009).
- [14] E. Fraś, A. Janas, A. Kolbus, M. Górny, Archives of Metallurgy 44(3), 253 (1999).

- [15] E. Fraś, A. Janas, P. Kurtyka, S. Wierzbiński, Archives of Metallurgy and Materials 48, 383 (2003).
- [16] A. Janas, A. Kolbus, E. Olejnik, Archives of Metallurgy and Materials 54(2), 319 (2009).
- [17] A.R.C. Westwood, Metallurgical Transactions A 19, 749 (1988).
- [18] C.F. F e n g, L. F r o y e n, Journal of Materials Science 35(4), 837 (2000).
- [19] Z.A. Munir, U. Anselmi-Tamburini, Metallurgical Transactions B 19, 155 (1988).
- [20] A.G. Merzhanov, Journal of Materials Processing Technology 56, 222 (1996).
- [21] E. Olejnik, A. Janas, A. Kolbus, B. Grabowska, Composites Theory and Practice **11(2)**, 120 (2011).
- [22] E. Olejnik, A. Janas, G. Sikora, T. Tokarski, J. Nowak, Archives of Foundry Engineering 13(1), 107 (2013).
- [23] E. Olejnik, M. Górny, T. Tokarski, B. Grabowska, A. Kmita, G. Sikora, Composite zones produced in iron castings by in-situ synthesis of TiC carbides, Archives of Metallurgy and Materials 58(2), 461 (2013).

Received: 15 February 2013.