Study of Microstructure of Oriented Eutectic Fe-C Alloy

M. Trepczyńska-Łent

Department of Materials Science and Engineering, Mechanical Engineering Faculty,
UTP University of Science and Technology, Al. prof. S. Kaliskiego 7, 85-796 Bydgoszcz, Poland
* Corresponding author. E-mail address: malgorzata.trepczynska-lent@utp.edu.pl

Abstract

Fe - 4,25% C alloy was directionally solidified with a constant temperature gradient of G = 33,5 K/mm and growth rate of v = 83,3 µm/s (300 mm/h) using a vacuum Bridgman-type crystal growing facility with liquid metal cooling technique. To reveal more detailed microstructure, the deep etching was made. This was obtained in the process of electrolytic dissolution. The microstructure of the sample was examined on the longitudinal and transverse sections using an Optical Microscope and Scanning Electron Microscope. Using the Electron Backscattered Diffraction technique, phase map and analysis of phase were made. In this paper the analysis of Fe-C alloy eutectic microstructure is presented. Regular eutectic structure was obtained. The fracture surfaces show lamellar structure. Microscopic observation after electrolytic extraction indicates that the grains of longitudinal shape of eutectic cementite have been obtained. These grains are characterized by layered construction with many rounded discontinuities.

Keywords: Directional solidification, Fe - C alloy eutectic, Microstructure, Longitudinal section, Transverse section

1. Introduction

During the liquid/solid phase transformation examined eutectic alloys formed extensive range of different phase microstructures. Eutectic alloys attract attention in the field of material science due to superior casting properties connected with a fine-scale composite microstructure.

Depending on the parameters which control the process, there are two basic techniques of solidification During isothermal solidification of pure materials, the solidification bath is kept at a constant temperature below the melting point, and the solid grows freely from this undercooled melt. While in directional solidification, a sample is being pulled with fixed rate through a fixed temperature gradient from a hot to a cold zone. Parametric study of these microstructures under well-controlled conditions is possible to perform because the microstructures formed in this process are very uniform [1, 2, 3].

Directional solidification is performed in a set-up shown in Figure 1. This is occasionally named as the Bridgman method. During this solidification one end is fixed to a cold zone and the other end to a hot zone. These zones are kept at fixed temperatures using a thermostat, respectively: below and above the melting point. This allows a fixed temperature gradient (G) to be established in the system. Between these zones the liquid metal is placed. As the solidification process progresses, solid phases grow in the direction of the hot zone at a particular growth rate (v) set by a motor pulling the sample towards the cold zone. These two parameters, G and v, are the main parameters that control directional solidification experiments. In common practice, the diffusion in the solid is ignored, as the solute diffusivity of solids is far smaller than the one of liquids. [4, 5].

For directional solidification, the growth rate is determined by the growth rate. It is important to determine the cooling of liquid at the front of solidification at a fixed growth rate.

For isothermal growth, the growth rate and characteristic dimensions of this obtained structure should be determined at a specified melt cooling. As there is a temperature gradient, the equilibrium concentrations at the solid-liquid interface are also functions of the coordinates at the interface. These concentrations are functions of temperature determined by the equilibrium phase diagram. This effect does not occur in isothermal growth and leads to a renormalization of both capillary effects and mass balance condition in the diffusion problem [7÷10].
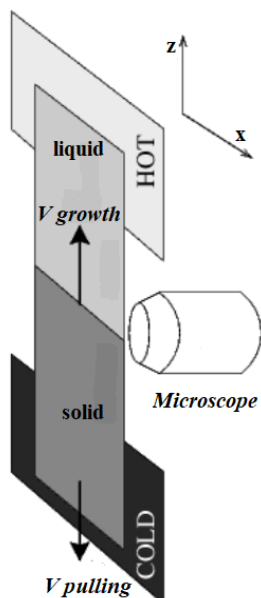

Fig. 1. Schematic of directional solidification [6]

The austenite-iron carbide eutectic $\gamma Fe + Fe_3C$ growth is initiated by forming of a cementite plate. The austenite dendrite nucleates and grows on this plate. There is destabilization around Fe_3C. Eutectic growth occurs: in the sidewise direction - a rod eutectic, and in the edgewise direction - a lamellar eutectic with Fe_3C as a leading phase. Cooling rate have a significant influence on the morphology of the γ-Fe + Fe_3C eutectic.

The metastable cementite is formed at~ $\approx$ 1147°C during cooling of the Fe-C system and starts to decompose at 500 °C during the heating process [11÷13].

2. Experimental procedure

2.1. Alloy preparation and directional solidification

The Fe - 4,25% C alloy sample was made from Armco and pressed graphite of spectral purity 99.99 %C. Under the protection of argon gas, in a corundum crucible in Balzers-type heater samples were made. They had diameter of 12 mm. After that they were machined to 5 mm in diameter.

This sample was loaded into Al_2O_3 tube of the vacuum Bridgman-type furnace. The sample was being heated to a temperature of 1450°C under an atmosphere of argon The sample was pulled out at a rate v=83,3 μm/s from the hot zone to the cold zone. The liquid metal alloy was used as the coolant (Fig. 1).

The specimen was grown by pulling it downwards. During that process a growth rate was fixed v=83,3 μm/s (300 mm/h) and a temperature gradient was constant G=33,5 K/mm [14, 15].

The process of directional solidification was made in the Department of Casting at the AGH University of Science and Technology in Cracow.

2.2. Metallography

The directionally solidified sample was cut, using electro-discharge machining, transversely and longitudinally, then ground flat through 600 grit paper and was polished with diamond paste. The sample was etched in a nital to reveal the microstructure.

To reveal more detailed microstructure, the deep etching was made in the process of electrolytic dissolution.

For the electrolytic isolation process of carbide (pro-eutectoid, eutectoid and eutectic cementite) As an electrolyte, 0.2N HCl was used. Electrolysis was made with the density of current of 5 to 6 mA/cm^2 during 5 hours [16].

This etched sample was examined using microscopy: optical (OM) and scanning electron (SEM).

EBSD technique phase map and analysis of phase were made. The Electron Backscattered Diffraction measurements were made in Institute of Metallurgy and Materials Engineering - Polish Academy of Sciences in Cracow.

3. Results and discussion

3.1. Microstructures

Figure 2 show the rod sample with marked growth direction.

Fig. 2. Researched sample

The fracture surfaces of directionally solidified eutectic alloy in the Figures 3 and 4 is represented. Figure 5 show microstructure on longitudinal section.

Figure 6 show EBSD measurements. Figures 7÷10 show transverse section of eutectic and Figures 8÷10 after the electrolytic isolation.
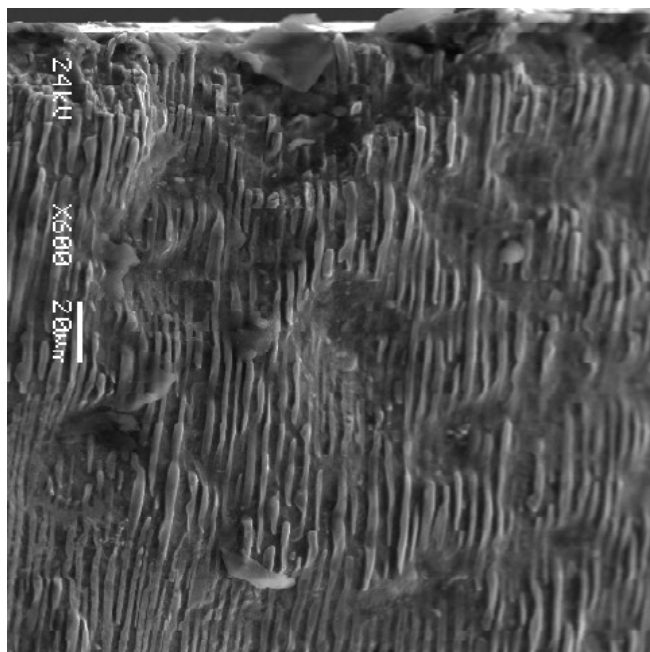

Fig. 3. Fracture surfaces of directionally solidified Fe-4,25%C composites with average lamellar spacing of 8,25 μm, SEM
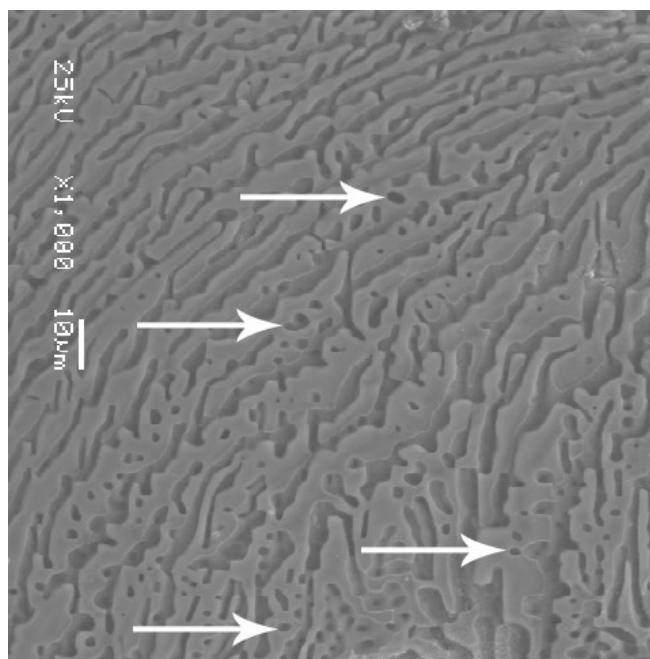

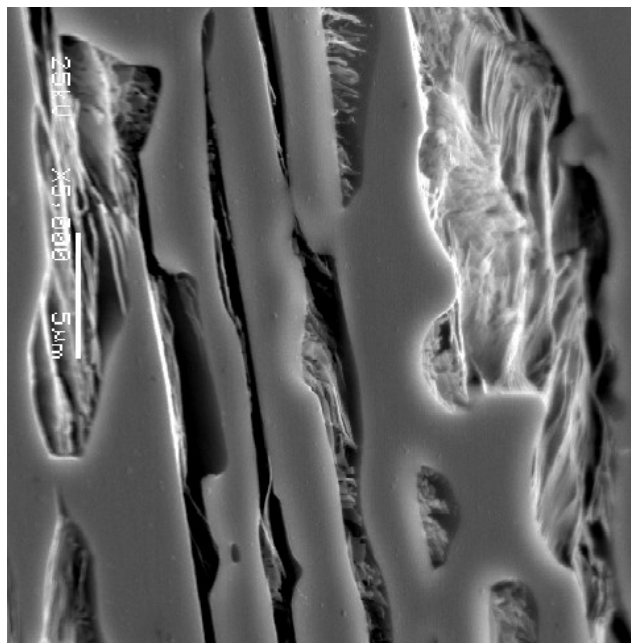Fig. 4. Fracture surfaces of directionally solidified Fe-C eutectic alloy, SEM

Fig. 5. Longitudinal section showing the microstructures of white eutectic, v=83,3 μm/s, G=33,5 K/mm, nital, SEM

The fracture surfaces (Figs. 3, 4) are provided, because they show the presence of lamellar structure very well. The lamellar cementite plates are parallel to each other and to the direction of the solidification. Lamellar Fe3C grow easily along the heat flow direction.

In Figure 4 fracture surfaces were probably formed due to too long and intense etching as one of preparation techniques. This led to the removal of the second component of the structure, which can be seen in Figure 5. In Figure 4, only one component of the structure - cementite - is visible. It has a lamellar shape with numerous discontinuities in the form of rounded, oval holes. In These discontinuities are indicated by arrows.

Micrographs showing lamellar microstructure along and throughout the cross-section of an alloy, directionally solidified, with composition Fe - 4,25% C are grown at 83,3 μm/s (300 mm/h) and G=33,5 K/mm.

In longitudinal section (Fig. 5), the longitudinal bright precipitates are visible. The direction of their alignment is in the line with direction of solidification. Between these precipitates there are fine structures.

EBSD studies (Fig. 6) show that this fine structure is a pearlite, while longitudinal bright precipitates are the iron carbide - cementite [17].

Figure 7 presents a transverse section of the microstructure at v =83,3 μm/s using Optical Microscope. Characteristic triangular areas were observed growing along the axis of the cylindrical sample. In these triangular areas, individual eutectic grains are parallel to each other.

The process of electrolytic dissolution revealed the existence of characteristic "agglomerates"- grains of cementite visible in transverse section. The Figure 8 shows that these grains adjust to each other through specific teeth. This can be seen precisely after the removal of pearlite after the electrolytic isolation (Figs. 8÷10).

Fig. 7. Microstructure of transverse section of white eutectic, v=83,3 μm/s, G=33,5 K/mm, magn. 50x, OM
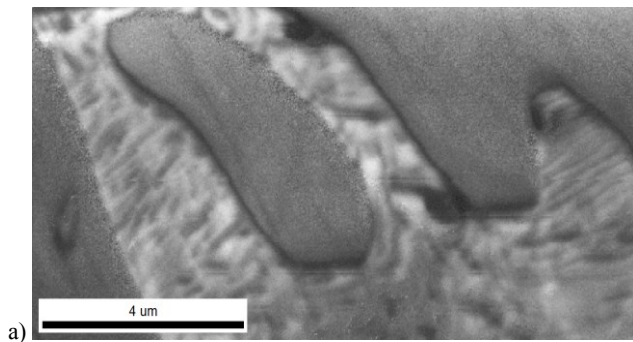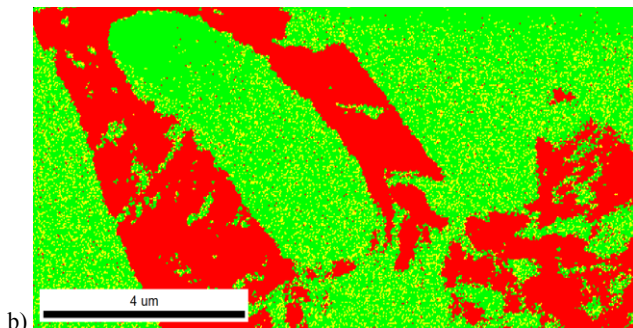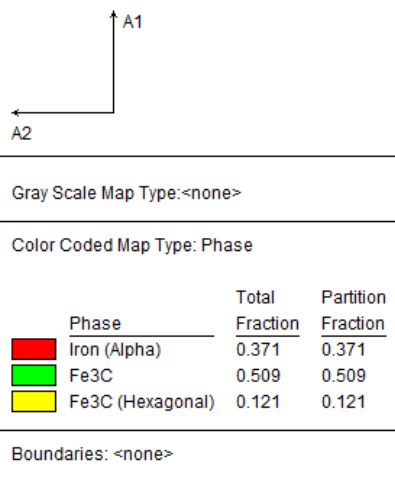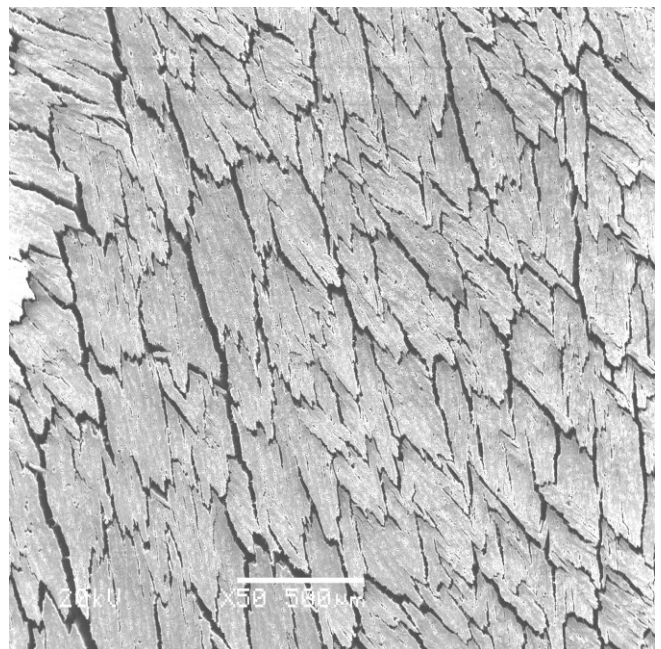
Fig. 6. Microstructure of white eutectic: a) SEM image, b) phase map (EBSD), c) analysis of phase (EBSD), v=300 mm/h

Fig. 8. Transverse section after electrolysis of eutectic, v=83,3 μm/s, G=33,5 K/mm, SEM

Inside the obtained fissures, layered growth of cementite (Figs. 9, 10) can be observed. This layers of cementite were formed inside visible "agglomerates – blocs". These blocks are separated by austenite fissures.

Cementite layers, also visible in Figure 4, are characterized by unsmooth surface – many discontinuities might be observed.
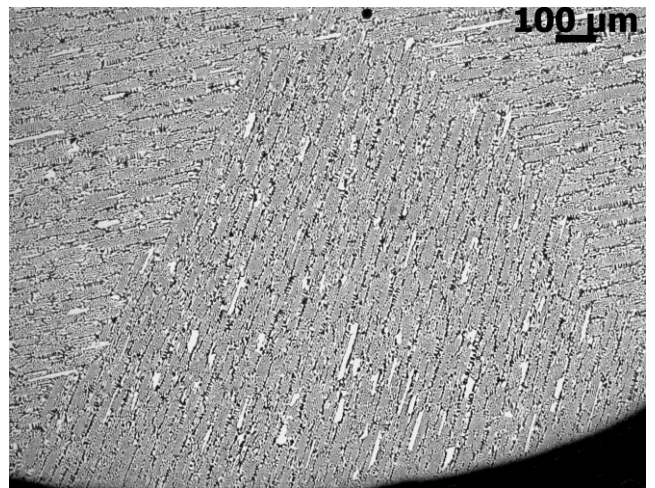
These discontinuities are where the austenite rods grew. Under the microscope only the transverse sections of the austenite rods can be observed. They are shown as dark, round-shaped figures on white substrate (cementite). After etching the iron phase is dark as it has decomposed to a fine pearlite structure.
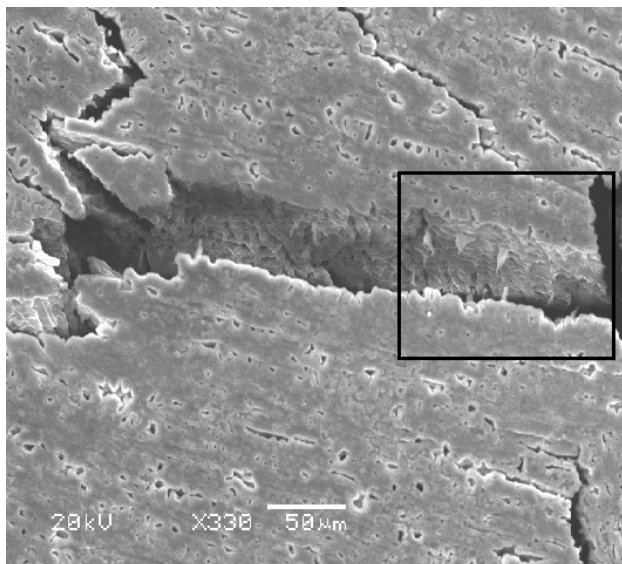
Fig. 9. Transverse section after electrolysis of eutectic,
v=83,3 μm/s, G=33,5 K/mm, SEM

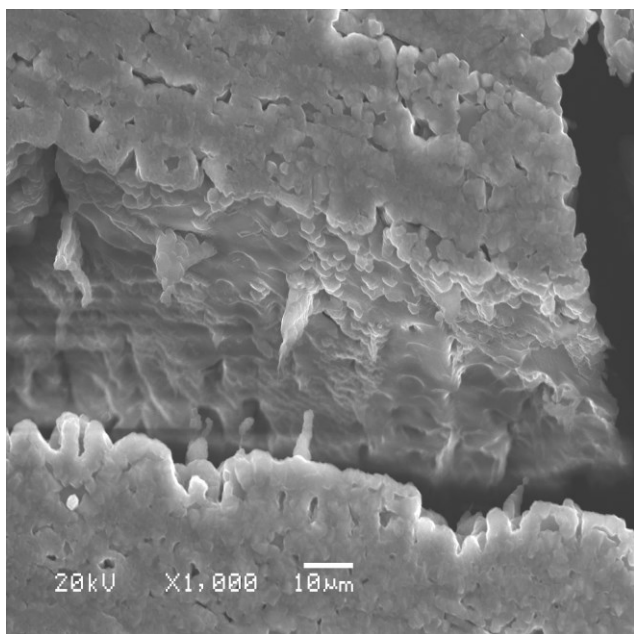Fig. 10. Transverse section after electrolysis of eutectic,
magnification of rectangle from Figure 9, v =83,3 μm/s, G=33,5
K/mm, SEM

4. Conclusions

Fe -4,25% C alloy was directionally solidified at a constant temperature gradient of G = 33,5 K/mm and growth rate of v = 83,3 μm/s. Regular eutectic structure was obtained.

Microscopic observation after electrolytic extraction indicates that the longitudinal grains of eutectic cementite have been obtained. These grains are characterized by layered construction with many round discontinuities.

Acknowledgements

References

[1] Kurz, W., Fisher, D.J. (1986). *Fundamentals of solidification.* v. 1. Trans Tech Publications.

[2] Stefanescu, D.M. (2009). *Science and engineering of casting solidification.* v. 2. Springer.

[3] Davies, G.J. (1973). *Solidification and casting.* Wiley.

[4] Utter, B. & Bodenschatz, E. (2002). Dynamics of low anisotropy morphologies in directional solidification. *Physical Review E.* 66:051604.

[5] Utter, B. (2001). Low anisotropy growth in directional solidification. *PhD thesis, Cornell University.*

[6] Ghosh, S. (2015). Effects of solid-solid boundary anisotropy on directional solidification microstructures. *HAL Id.*

[7] Brener, E.A. & Temkin, D.E. (1996). Cellular, dendritic, and doublon patterns in directional crystallization. *JETP.* 82(3).

[8] Wołczyński, W. (2000). in: Modelling of transport phenomena in crystal growth. *J.S. Szmyd & K. Suzuki, Ashurst Lodge,* Southampton, UK - Boston, USA. 19.

[9] Piątkowski, J. & Matuła, T. (2012). Estimation of the operational reliability determined with Weibull modulus based on the abrasive wear in a cylinder-piston ring system. *J. of Achievements in Materials and Manu-facturing Engineering.* 55(2), 416-420.

[10] Stefanescu, D.M. (2005). Solidification and modeling of cast Iron—A short history of the defining moments. *Materials Science and Engineering A.* 413-414, 322-333.

[11] Nastac, L. & Stefanescu, D.M. (1995). Prediction of gray to white transition in cast iron by solidification modelling. *AFS Transactions.* 103, 329-337.

[12] Schumann, H. (1983). *Metallographic 11. Auflage,* VEB Deutscher Verlag fur Grundstoffindustrie, Leipzig, 314.

[13] Kopyciński, D. (2013). The inoculation of white cast iron. TMS (The Minerals, Metals & Materials Society). *Published by John Wiley&Sons, Inc., Hoboken,* New Jersey. Supplemental Proceedings. 601-608.

[14] Trepczyńska-Łent, M. (2017). Directional solidification of Fe-Fe$_3$C white eutectic alloy. *Crystal Research and Technology.* 52(7), Version of record online: 26 JUN 2017. DOI: 10.1002/crat.201600359.

[15] Trepczyńska-Łent, M. (2013). Possibilities of the materials properties improvement for the cementite eutectic by means of unidirectional solidification. *Archives of Metallurgy and Material.* 58(3), 987- 991.

[16] Kitagawa, H., Shibata, N. (1961). Electrolytic isolation of carbide, sulphide and phosphide in white cast iron. *Transactions of the Jap. Inst. of Metals*. 2(2), 130-133.

[17] Trepczyńska-Łent, M. (2016). XRD and EBSD measurements of directional solidification Fe-C eutectic alloy. *Archives of Foundry Engineering*. 16(4), 169-174.