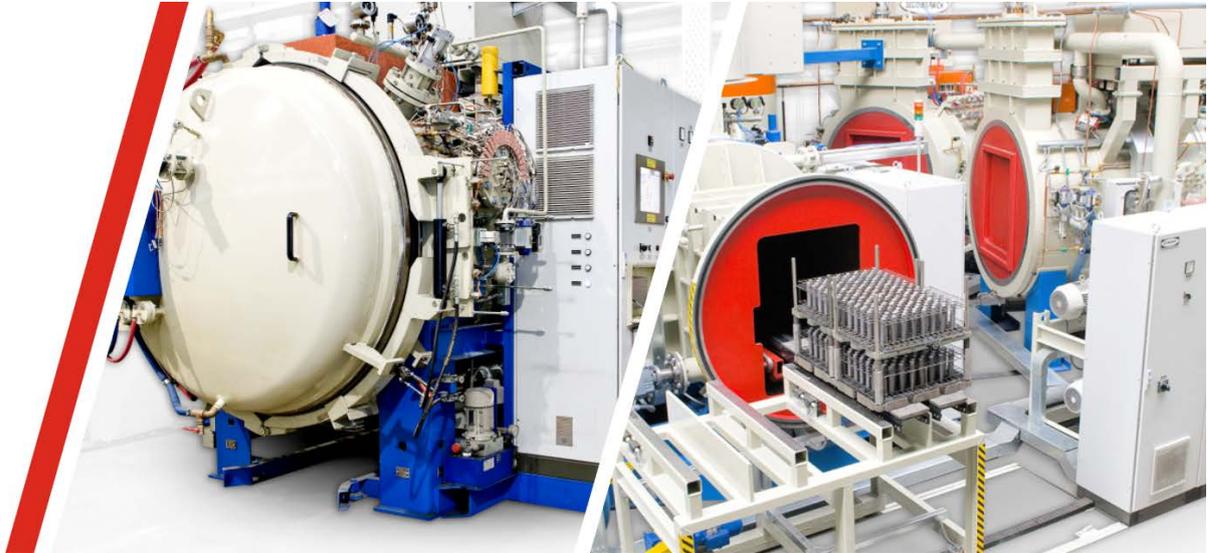


MODERN HEAT TREATMENT OF FERROUS ALLOYS WITH AN ISOTHERMAL STOP

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INTRODUCTION

High mechanical properties of steel are most often obtained by heat treatment, specifically hardening and tempering. The martensitic hardening process dramatically increases the hardness and strength of steel, but at the same time it reduces plasticity and brittleness. In addition, it produces high stress in the material and, consequently, changes the dimensions of the hardened element. In order to improve the plasticity, the tempering process is applied after hardening, although increasing the tempering temperature increases the plasticity but decreases the strength. Unfortunately, the tempering process does not reduce hardening deformations.

A good combination of high hardness and mechanical properties as well as impact strength and plasticity can be achieved through the use of the thermal treatment, isothermal quenching. This treatment, unlike continuous hardening with martensitic transformation, most often uses bainitic transformation. The isothermal quenching is based on austenitization followed by rapid cooling to bainitic transformation temperature and remaining at this temperature for the time needed to complete the bainite conversion and final cooling to ambient temperature (Figure 1). It allows the formation of a lower bainite microstructure, comprising of carbon oversaturated bainite ferrite slabs with inclusions of cementite. In ADI (Austempered Ductile Iron) cast iron, this treatment is used to

produce ausferrite, a mixture of plate ferrite and residual austenite. Such microstructures provide greater plasticity, ductility and impact resistance and less hardening deformation than martensite. However, the above benefits are diminished by lower values of strength parameters and limited hardness (max 55 HRC). Moreover, in order to rapidly cool to the temperature of the isothermal stop, non-ecological and hazardous molten mineral baths are usually used.

As a result of isothermal quenching, comparable and even better mechanical properties with higher plasticity and fracture toughness than after continuous quenching, is possible by creating the microstructure of nanobainite noncarbide [1 - 5]. Conducting an isothermal interval under precisely defined time and temperature conditions in high carbon and silicon and/or aluminum alloys results in a unique microstructure consisting of bainite-thickened ferrite carbonaceous plates with a thickness of less than 100 nm surrounded by a thin layer of residual austenite (Figure 2). Strong structural fragmentation and alternate arrangement of hard bainitic and plastic austenite ferrite provide high strength and crack resistance while maintaining hardness comparable to low tempered martensite.

EXAMINED MATERIALS AND METHODOLOGY

The range of materials used in research on nanobainite manufacturing technology included various steel grades available on the Polish market, spheroidal cast iron castings with various alloying additives, and experimental cast steel castings designed for nanostructures. The chemical composition of the materials studied in this paper is shown in Table 1.

Some of the steels (38CrAlMo6-10 and 35CrSiMn5-5-4) under investigation were selected due to the low carbon content in relation to the content of which, according to Bhadeshii and contributors [6] should consist of nanostructured steels, were subjected to vacuum carburization by injection method, FineCarb® technology according to the SECO/WARWICK patent. [7]

Heat treatment with isothermal quenching was carried out in laboratory furnaces at the Warsaw University of Technology. Austenitization was carried out in a gas-tight oven in a nitrogen-protective atmosphere. The isothermal quenching processes were carried out in a tin bath and a fluid bed at temperatures of 320-250 °C. The times of the isothermal stop were chosen to bring about the end of bainitic transformation in a given material. Some samples of 100CrMnSi6-4 steel were subjected to austenitization and hardening by gas (compressed nitrogen) tempering to isothermal temperature in a SECO/WARWICK vacuum furnace.

Thin film observations were made using the IMI 1200 electron microscope, operating at 120 kV.

Tensile strength tests were carried out in accordance with PN-91/H-04310 on round specimens of ten times. The static tensile test was performed on a Zwick/Roell Z250 endurance machine using an MTS extensometer.

Impact tests were performed in accordance with EN ISO 148-1_2011 using a Charpy Zwick/Roell RKP 450 hammer with 300J power. The tests were performed on 10 x 10 x 55 mm V-notched specimens.

The friction wear test was carried out using a roller-block method according to ASTM G77 using a T-05 tester, under 200N and 400N load on 6.35mm wide rectangular specimens. As the counter was used, the outer diameter of the rings was 34.99 mm, made of steel. 100Cr6 bearings with a hardness of 62 HRC.

PROPERTIES AND MICROSTRUCTURE OF STEEL AFTER ISOTHERMAL QUENCHING

As far as project Nanostal [8] is concerned, it has developed the Nanostructural technology of selected grades of commercial steel, providing a number of unique properties. Correctly selected isothermal tempering parameters allowed for a combination of high strength combined with good plasticity for both structural and tool steels. For example, for the X37CrMoV5-1 tool steel after nanobainity, a tensile strength of 1860 MPa was obtained at 16% elongation. In 67SiMnCr6-6-4 bearing steel with a tensile strength of 1500 MPa, the elongation to break was 25%.

Nanocitalization tests in an industrial furnace carried out on 100CrMnSi6-4 steel have shown that it is possible to scale technology from a laboratory to an industrial scale [9]. The treatment of the isothermal quenching of this steel in a vacuum oven resulted in the formation of nanobainite with an average thickness of approximately 30 nm (Figure 3). The tensile strength of the treated steel was 1780 MPa at 7% elongation, KV 9 J impact strength.

Nanostal [8] and Nano4Gears [10] studies have also been able to develop an isothermal quenching technology to produce nanobainite in the steel top layers previously subjected to carburization. The isothermal stop parameters are then selected so that the nanobainite microstructure is formed in the carburized surface. Examples are two 38CrAlMo6-10 and 35CrSiMn5-5-4 steels, vacuum carburized then isothermal tempered. The treatment of isothermal quenching resulted in the formation of nanobainite microstructure in the carburized layer and tempered martensite in the core of the steel tested (Figure 4). Carrying out the T-05 wear test showed that the nanobainitic layers exhibited several times less volume loss as a result of abrasion than the martensite structure produced in the same steels

(Figure 5) [11]. The reason for this behavior of nanobainitic steels is the TRIP effect that occurs in the residual austenite on the surface while the steam is working.

FOUNDRY IRON ALLOYS

Ductile cast iron, containing an average of 1 - 3% silicon, has been subjected to isothermal quenching for many years to achieve the optimum combination of strength and plasticity. The presence of silicon results in the formation of ausferritic microstructure instead of bainite, a mixture of plate ferrite and austenite. The research [12] conducted within the NanoDI project has shown that as a result of carefully selected parameters in spheroidal cast iron, the fragmentation of the microstructure can be achieved to a nanometer scale.

An example is spheroidal copper with a pearlite matrix and the chemical composition given in Table 1 (ADI 1). Cast iron was subjected to isothermal tempering at two different temperatures: 280 and 300°C. As a result of the treatment, a tensile strength of more than 1500 MPa was obtained - after isothermal quenching at 280°C, with an elongation of about 1%. As a result of the heat treatments performed at both isothermal temperatures, a significant improvement in impact was observed compared to the initial state. The highest impact strength was observed for samples after isothermal tempering at 300°C.

Another example of nanostructured cast iron is nickel-molybdenum cast iron, with the chemical composition given in Table 1 (ADI 2). The microscopic observations carried out after the isothermal quenching process revealed a microstructure consisting of thin ferrite plate packages separated by residual austenite layers (Figure 6). Residual austenite was also observed in the form of blocks with an average size of about 3.9 μm^2 . The total share of austenite in the structure was about 22%.

The formation of nanostructure in ADI 2 cast iron resulted in increased hardness and tensile strength compared to the conventional ausferrite microstructure (Figure 7). The plasticity and impact strength after nano-structurisation have been found to be lower, possibly due to the lower residual austenite content. Adequately high proportion of austenite blocks allows for the appearance of TRIP effect during deformation, thereby increasing the plasticity of the material. Significant fineness of the microstructure in nanobainiting cast iron contributes to increased strength, while reducing the number and average size of residual austenite blocks reduces TRIP effect, negatively affecting the plastic properties of cast iron.

The experimental melting heat of the isothermal hardening (Figure 8) was characterized by a tensile strength of 1760 MPa at 1.4% elongation and 30 J impact strength (specimens without score).

SUMMARY

The research has shown that it is possible to produce a nanocrystalline structure in the commercially available grades of steel and ductile cast iron by means of the relatively simple heat treatment of isothermal quenching. This is the first cheap and efficient nanostructurization technology that allows for massive and complex geometry. The developed technology can be realized using existing heat treatment equipment. Tests undertaken in SECO/WARWICK furnace have shown that conducting isothermal quenching to nanobainite is also possible in vacuum technology, which eliminates the need for environmentally damaging saline baths.

Thanks to the use of nanostructurization technology, it is possible to obtain better mechanical properties of steel compared to thermal improvement. The combination of high strength and good plasticity, comparable to the properties of expensive *maraging* steel, thanks to nanobainiting technology, is possible to achieve in easily accessible and affordable low and medium alloy steels. In addition, nanobainite exhibits much better wear resistance than martensite with the same or higher hardness. The unique properties of nanobainitic steels allow for application in a wide range of applications from high-strength bolts and fasteners, engine elements and turbine to arms and armor elements.

Hardening with an isothermal stop on the formation of nanobainite spheroidal cast iron provides higher hardness and mechanical properties compared to traditional ADI cast iron. Higher durability parameters may be important when using cast iron in the mining industry for the construction of mining machinery and transmission components.

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Project PBS III No. 246715 "Developing an innovative, casting-based approach, Technology of manufacturing nanocrystalline gear wheels "

Project No POIR.04.01.04-00-0064 / 15-00, 2017r. " Development of innovative technology for producing toothed components with hybrid nanostructured surface coatings for conveyor drive units designed for extreme operating conditions. "

LITERATURE

- [1] Caballero F. G., Badeshia H. K. D. H., Mawella K. J. .: Very strong low temperature bainite, *Mat. Sci and Technol.* 18 (2002), 279 ÷ 284
- [2] Garcia-Mateo C., Caballero F. G., Badeshia H. K. D. H. .: acceleration of low-temperature bainite, *ISIJ Int.* 43 (2003) 1821 ÷ 1825
- [3] Garcia-Mateo C., Caballero F. G., Badeshia H. K. D. H. .: Development of hard bainite, *ISIJ Int.* 43 (2003) 1238 ÷ 1243
- [4] Dharamshi H. K., Badeshia H., K. D. D., Garcia-Mateo C., Brown B.: Bainite Steel and Methods of Manufacture, US Patent Application Publication, No. 2011/0126946 A1, 2 June 2011
- [5] Świątnicki W.A., Pobiedzińska K., Skołek E., Gołaszewski A., Marciniak Sz., Nadolny Ł., Szawłowski J.: Otrzymywanie struktury nanokrystalicznej w stalach przy wykorzystaniu przemiany bainitycznej, *Inżynieria Materiałowa*, rok XXXIII, nr 6 (2012) 524-529
- [6] Dharamshi H. K., Badeshia H. K. D. H., Garcia-Mateo C., Brown P.: Bainite Steel and Methods of Manufacture Thereof, Patent Application Publication, No. US 2011/0126946 A1, 2 June 2011
- [7] Kula P., Olejnik J., Heilman P.: European Patent No. : EP1558780 (2007), United States Patent No.:US 7513958 (2009)
- [8] Final report of the project: " Wytwarzanie stali o strukturze nanokrystalicznej przy wykorzystaniu przemian fazowych" project no WND-POIG.01.01.02-14-100 / 09, 2015r.
- [9] Dworecka J., Jezierska E., Roźniatowski K.: Characterization Of Nanobainitic Structure Obtained In 100CrMnSi6-4 Steel After Industrial Heat Treatment, *Arch. Metal. Mat.* 59 (2014) 1637 – 1640
- [10] Report on the project "Opracowanie innowacyjnej technologii produkcji elementów uzębionych z hybrydowymi warstwami powierzchniowymi o podłożu nanostrukturalnym do zespołów napędowych przenośników przeznaczonych do pracy w ekstremalnych warunkach eksploatacyjnych", No POIR.04.01.04-00-0064 / 15- 00, 2017r.
- [11] Wasiluk K., Wasiak. K., Skołek E., Świątnicki W .: Formation of Nanobainitic Structure in Carburized Layer of Structural Steels, *Metal 2014*. Tanger Ltd, Ostrava, Czech Republic, 1 - 7
- [12] Mice D., Wasiluk k., Skołek E., Świątnicki: Nanoausferritic matrix of ductile iron. *Materials Science and Technology*, vol. 31, no 7 (2015) 829-834.

Table 1. Chemical composition of the materials tested

	C	Si	Mn	Mg	Ni	Mo	Cu	Cr	Al	V	P	S
X37CrMoV5-1	0,36	1,00	0,35	-		1,3		5,5		0,4		
67SiMnCr6-6-4	0,67	1,55	1,40	-	-	0,25	-	1,10	-	-	0,02	0,015

100CrMnSi6-4	1,00	0,60	1,10	-	-	-	-	1,55	-	-	0,01	-
38CrAlMo6-10	0,40	0,32	0,65		0,20	0,25	0,16	1,54	0,99	-	0,017	0,003
35CrSiMn5-5-4	0,35	1,30	0,95	-	0,14	-	0,15	1,31	-	-	0,012	0,01
ADI 1	3,44	2,32	0,28	-	-	-	1,13	-	-	-	-	-
ADI 2	3,50	2,54	0,16	0,047	1,4	0,24	0,5	0,03		-	0,04	0,013
Cast	0,67	1,33	0,609	-	1,54	0,31	-	0,50	-	-	0,020	0,018

Table 2. Selected mechanical properties of the tested alloys after nanobainity

Material	X37CrMo V5-1	67SiMnCr 6-6-4	100CrMnS i6-4	ADI 1	ADI 2	Cast
HRC	52,5	49,7	52	41,1	37,2	53,6
R _{0.2} [MPa]	890	1070	1370	1100	1030	1290
R _m [MPa]	1860	1540	1780	1330	1220	1760
A ₅ [%]	16	25	7	2	4,3	1,4
KV [J]	15	18	9	3,9	9	29

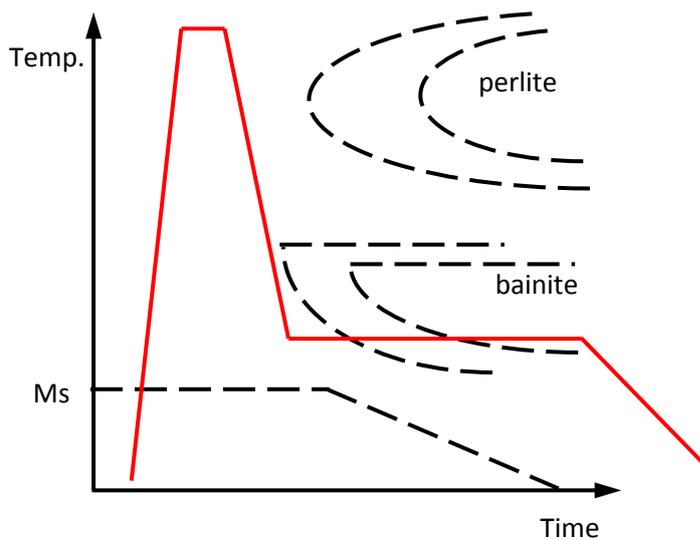


Figure 1. Schematic diagram of heat treatment of isothermal quenching.

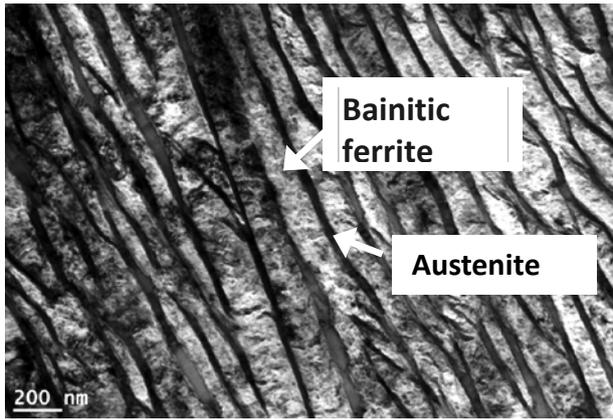


Figure 2. Nanobainite microstructure, TEM image.

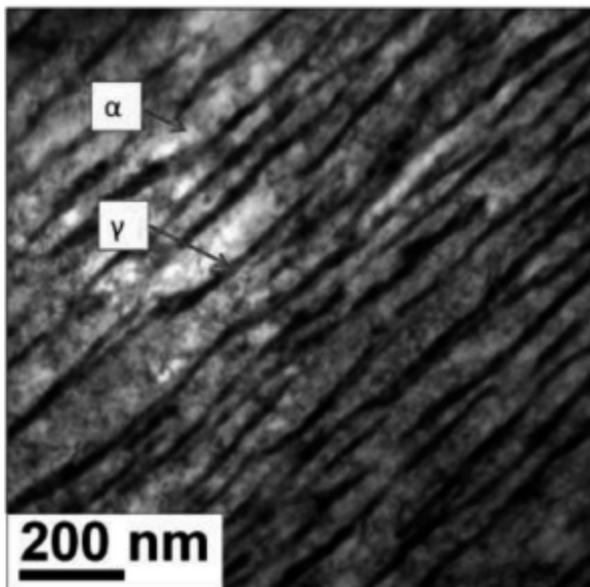


Figure 3. Microstructure of 100CrMnSi6-4 steel after nanobainitization in a vacuum oven, TEM image.

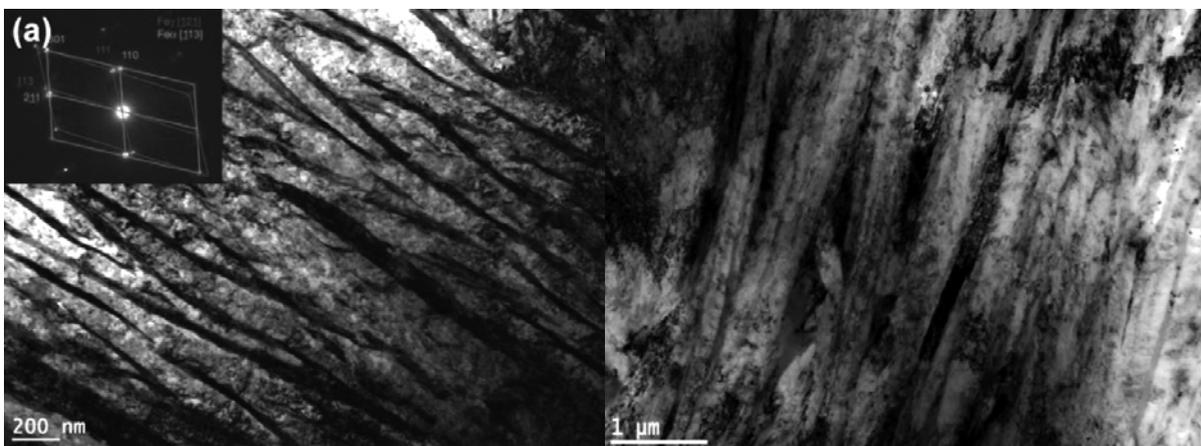


Figure 4. Microstructure of 35CrSiMn5-5-4 steel after carburization and nanobaiting - a) coarse layer, b) core. TEM image

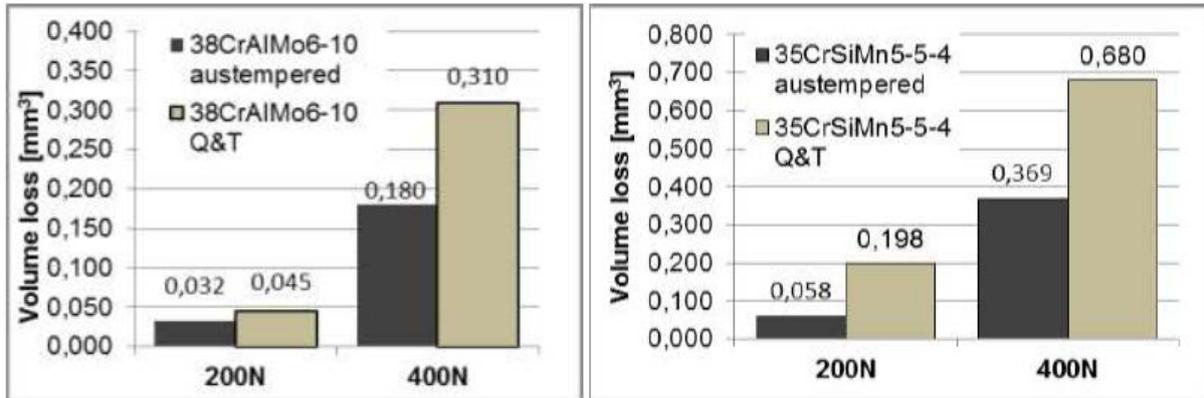


Figure 5. Comparison of volume losses after t-05 friction tests of 38CrAlMo6-10 and 35CrSiMn5-5-4 steels after austempered nanobaiting and after tempering and low tempering (Q&T)

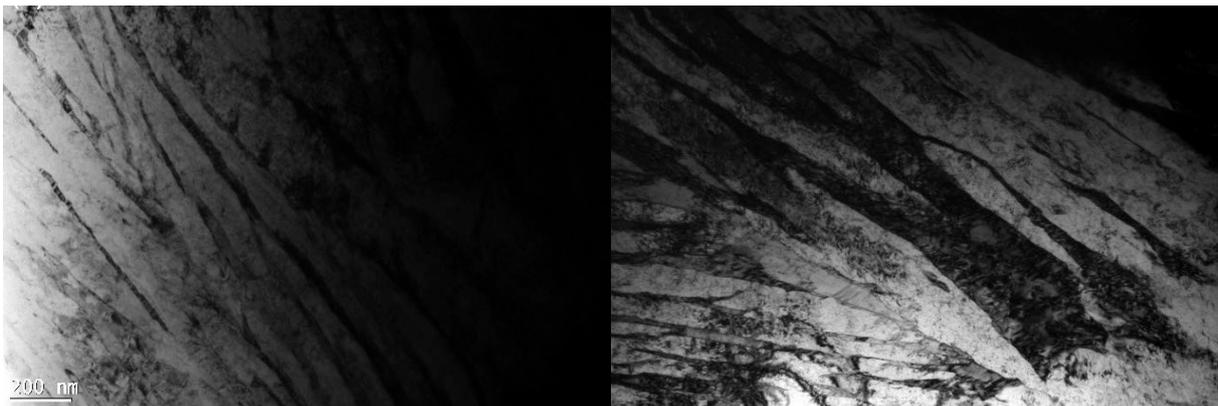


Figure 6. Microstructure of ADI 2 cast iron after isothermal tempering, TEM image

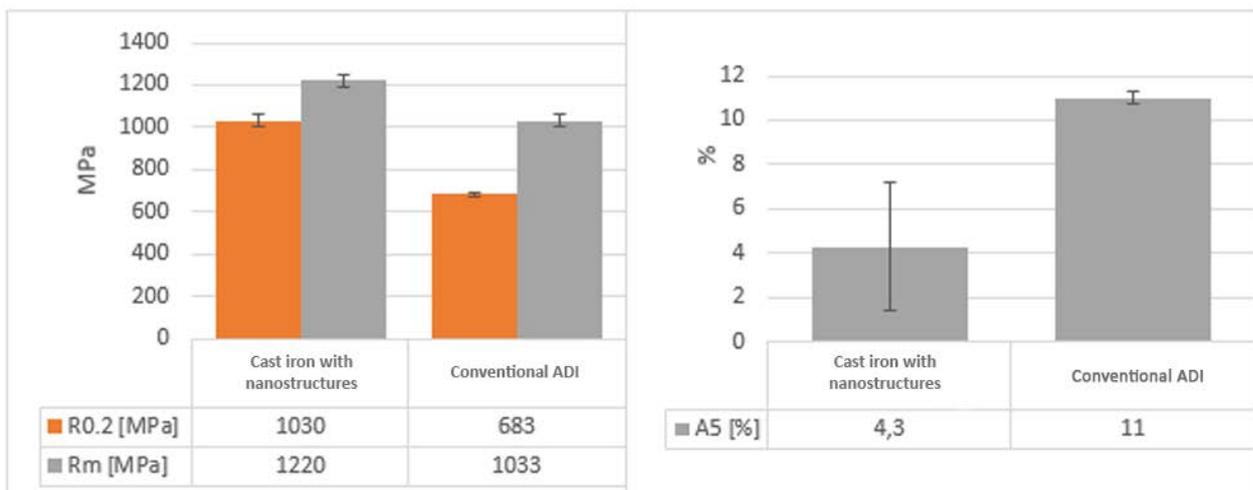


Figure 7. Comparison of ADI 2 cast iron properties after isothermal quenching to nanostructured and conventional



Figure 8. Microstructure of cast steel after isothermal quenching, TEM image