Nucleation and graphite growth in nodular cast iron – an overeview

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Humans - Valuable Resource for Foundry Industry Development

Split, June 16th – 18th, 2021

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Humans - Valuable Resource for Foundry Industry Development

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PREFACE

Knowledge is becoming an increasingly important resource for economic development. The Republic of Croatia is facing the challenges of the world economy, with the aim to meet certain requirements in shaping the education system. Ensuring the quality assurance of the education system is just one of the requirements set up as a continuous mission of University of Zagreb Faculty of Metallurgy and other co-organizers from the high-education. As the level of education of the population affects the progress of the economy, it is extremely important for the Republic of Croatia to increase the ratio of highly educated persons. In recent years, the ratio of the highly educated population of the Republic of Croatia has been growing, but in comparison with Europe, Croatia is still lagging behind. In order to increase the share of highly educated persons, it is necessary to invest in the quality of education, both in higher education and in secondary and primary education. This would increase awareness of the importance of education, which would ultimately result in an increase in the ratio of highly educated and competent professionals.

Metal industry as a base branch represents an important factor contributing to the economic potential of each country. Current market development as well as technical and economic objective, the production of high-quality, low-cost and environmentally friendly casting, requires application of recent and advanced materials, as well as production technologies, followed and supported by understanding of production process. The metal industry has been recognized as a "driving subdivision" of economy development.

Until the recession and deepening of the economic crisis in Croatia, companies operated stably, focused on streamlining production, investing in technology and employee's education, increasing product quality and productivity, developing innovation and fighting for the market. The recession and economic crisis have slowed the strengthening of this economic activity. In order to overcome and mitigate the negative results caused by falling orders and reduced production, companies have developed new production programs and sought new customers and markets in order to maintain good positions within their market niches. Taking into account the growing need of large (global) producers for small series products, it is assumed that it will build a network of suppliers in which Croatian producers can be included. Small quantities are sufficient to employ their production capacities, and with a skilled workforce and new market opportunities, the growth of existing companies is expected, as well as the establishment of new ones. By investing in modern equipment and production certification, metal producers indicate a desire for growth. The main features of Croatian industry are stable product quality and reliability in accordance with EU standards, while on the other hand it is important to invest in available professional workforce, targeted support of scientific institutions, good production infrastructure with emphasis on modern technologies and transport links to the world.

Despite the recognizability and importance of the profession, the profession is underestimated by the amount of the average net monthly salary per employee in legal entities. The gross value added of the product is also indicative. Since the Croatian market is too small for significant production growth, companies in the observed activity primarily direct their production capacities to EU countries, which also means increasing the level of productivity of assets and labor. Competitiveness can be based exclusively on modern technology, efficient production processes but also on a highly skilled workforce. All this requires investment in infrastructure and educational study programs that should strive to acquire primarily practical knowledge and skills with an emphasis on the development and application of modern materials and technologies, in order to change this status of the Republic of Croatia.

Therefore, the motto of the ***19th International Foundrymen Conference*** is focused to the **HUMANS** as a **valuable resource for foundry industry development**. Human resources have an unavoidable role in scientific, technological and practical aspects concerning research, development and application of casting technology with the common perspective – increase of competitiveness.

Special attention will be focused towards the competitiveness ability of foundries, improvement of materials features and casting technologies, environmental protection as well as subjects connected to the application of castings.

During this Conference 49 papers will be presented in hybrid mode (online and in situ) due to pandemia of COVID-19 virus. In this Conference scientists from 14 countries (Australia, Austria, Bosnia and Herzegovina, Croatia, Czech Republic, India, Kosovo, Poland, Romania, Spain, Serbia, Slovenia, Slovakia, United States of America) recognized the importance to be a part of this scientific event. Book of Abstracts of the 19th International Foundrymen Conference includes summaries of the papers. The Proceedings book consists of papers *in extenso* published in electronic format (USB). Full length papers have undergone the international review procedure, done by eminent experts from corresponding fields, but have not undergone linguistic proof reading. Sequence of papers in Proceedings book has been done by category of papers in following order: plenary lectures, invited lectures, oral and poster presentation, and inside the category alphabetically by the first author's surname.

Within the Conference Student section is organized. This is an opportunity for industry to meet and recruit human resources as a main potential for business development. Coexistence of material science and sustainable technology in economic growth represent a knowledge transfer between small and medium enterprises' (SMEs´), industry and higher education institutions. Higher education at the Faculty of Metallurgy (HEI), conceived through the program and the learning outcomes, is based, inter alia, on promoting students' scientific and research work on applied topics, enabling ambitious and creative young people to become independent problem solvers, developing and supporting their curiosity, analytics and communication: **Graduates like the labour market needs!**

This occasion represents an opportunity to discuss and increase the mutual collaboration between HEIs' and industry with the aim of information exchange related to advanced experience in foundry processes and technologies, gaining the new experience in presentation and / or teaching methods and techniques within lifelong learning process.

The organizers of the Conference would like to thank all participants, reviewers, sponsors, auspices, media coverage and all those who have contributed to this Conference in any way.

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NUCLEATION AND GRAPHITE GROWTH IN NODULAR CAST IRON - AN OVEREVIEW

Barbara Tubić Bulat[1*], Zdenka Zovko Brodarac[1], Primož Mrvar[2]

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Oral presentation
Subject review

Abstract

The term nodular iron cast refers to a group of iron alloys with a maximum carbon content of 4 wt.% and silicon from 1.7 wt.% to 2.8 wt.%. This type of cast iron is characterized by the presence of carbon in the spherical or nodular form of graphite in a ferrite, pearlite or ferrite-pearlite matrix. Due to its better mechanical properties in relation to the other iron cast groups, it is widely used in production of gearboxes, crankshafts, turbine rotors, tanks, transportation industries, etc. Generally, size, distribution and shape of graphite nodules ensure a good toughness, high value of elongation and yield strength, castability and machinability.
The course of solidification and each parameter of the production and solidification process has a certain impact on the mechanical properties and microstructure of the final product. However, solidification and graphite growth mechanisms in nodular cast iron have not been fully explained in despite to a large amount of research on the subject. Different experiments have led to the creation of different theories about the solidification process which aim to enable the creation of models for predicting the solidification process and the growth of graphite nodules. Predicting the development of microstructure and defining optimal parameters of the production process could prevent casting and microstructural defects thus ensuring desired properties of the casting.
This paper provides an overview of several studies regarding the nucleation and graphite growth mechanism theories and experiments.

Keywords: *casting, nodular iron, solidification, nucleation, graphite growth, overview*

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INTRODUCTION

Prior to the use of steel in construction industry, cast iron was the most common material for such application. Continuous progress of research, casting industry development and lower production costlead to the revival of iron castings as desirable construction materials [1]. Microstructure is a significant element of physical metallurgy of iron alloys and

represents the relationship between chemical composition, production processes, microstructure and mechanical properties.

The Fe-Fe_3C diagram (Figure 1) shows a map of the phases present in iron alloys under equilibrium conditions relating the ratio of carbon content to the temperature. Equilibrium assumes that there is enough time available to achieve the right chemical compositions for phases to form [2]. Silicon is used in the industrial production of ferrous alloys to achieve the so-called carbon equivalent which is calculated according to equation 1.

$$CE = \% C + 1/3 \% Si + 1/3 \% P \quad (1)$$

Carbon equivalent defines a type of alloy. Thus eutectic alloy's CE equals 4.3 % while hypereutectic alloys have CE above 4.3 or CE lower than 4.3 for hypoeutectic alloys respectively [3].

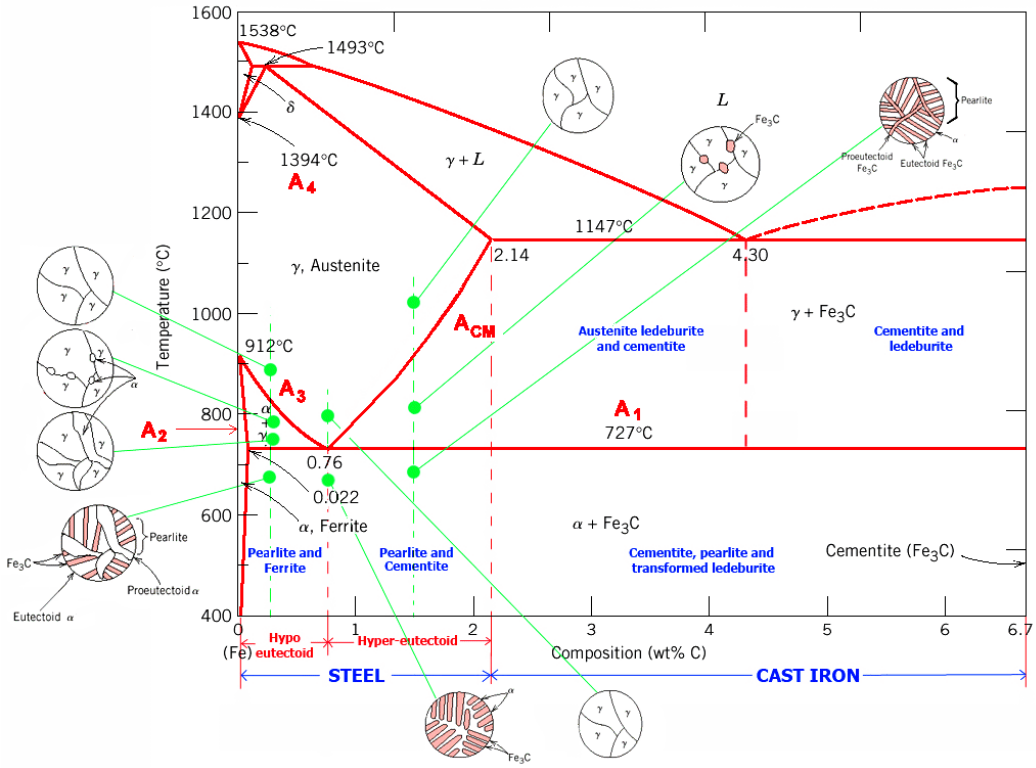

Figure 1. Iron- carbon phase diagram [4]

Nodular cast irons are a group of iron alloys containing a maximum 4 wt.% of carbon and 1.7 to 2.8 wt. % of silicon. Such alloys are characterized by the presence of carbon in spherical or nodular form of graphite in the ferrite, pearlite or ferrite-pearlite metal matrix [1]. The nodular form of graphite is achieved by magnesium and rare earth elements (RE) addition to the liquid iron before or during casting process [5]. This contributes to the improvement of mechanical properties such as strength, castability and toughness of cast iron. Nodular iron has mechanical properties comparable to those of some high-carbon steels achieved at lower production cost. Due to its better mechanical properties in relation to the other iron cast groups, it is widely used in production of gearboxes, crankshafts, turbine rotors, tanks, transportation industries, etc. [6]. Figure 2 shows the interdependence of the relative mass(a) and the relative production cost (b) of different materials products with tensile

strength in the product. Nodular cast iron has a very favorable ratio of price and mechanical properties in comparison to other metal materials which discloses its´ significant application.

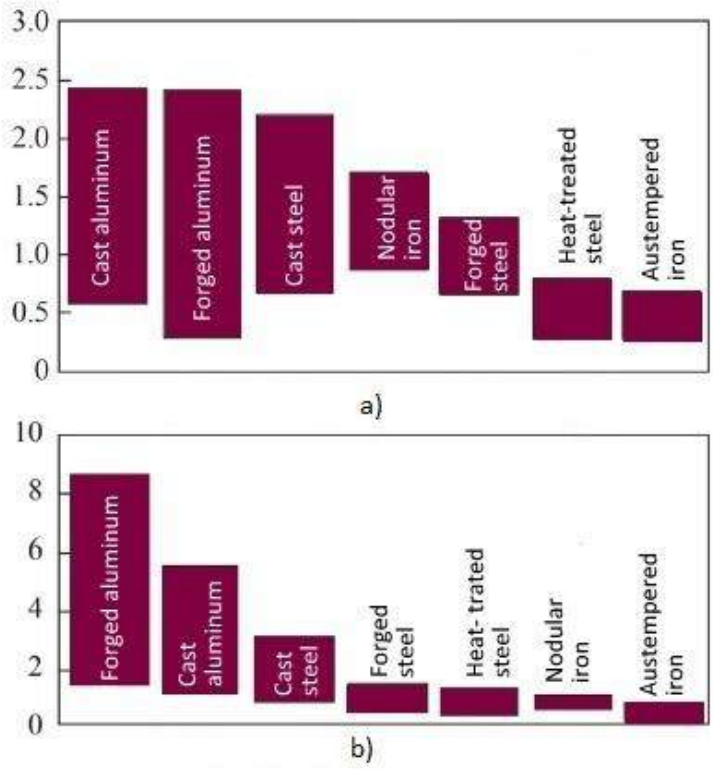

Figure 2. Comparison of
a) weight of different materials in relation to strength
b) relative cost of different materials in relation to strength [7]

Nodular iron castings are iron alloys used industrially in parts that require moderate plasticity without sacrificing mechanical resistance, such as valves, hardware elements, and auto parts. Studies focused on nodular iron castings have become important in recent years due to its extensive use and the growing trend of replacing forged steels due to lower production costs [8]. The improvement of the mechanical properties of ductile iron castings is achieved by alloying, which enables the replacement of cast and forged steel with nodular cast irons in various applications. In order to achieve further improvements in the quality of ductile iron casting parts, the physical mechanisms that make up the solidification process have to be explained. The mechanical properties of ductile iron castings are influenced by the metal matrix and morphology of graphite nodules [8, 9]. The solidification process of ductile iron starts at a temperature of 1147 °C (Figure 1), which makes it difficult to collect experimental data on the solidification sequence. The latter is therefore described assuming the cooling process in the liquid state of a well-insulated casting takes place in equilibrium conditions where the cooling rate is equal to zero. The first solid phase developed in hypereutectic (carbon equivalent CE = 4.5%) is a graphite cluster phase [8, 10]. Clusters are graphite particles that grow by binding carbon atoms from the melt until the temperature reaches the eutectic reaction temperature. Then in areas with low carbon concentration austenite is formed which then surrounds the graphite nodules. During eutectic solidification, only austenite is in direct contact with the melt. The further growth of graphite nodules takes place through austenite by carbon diffusion [11, 12]. After solidification ends, carbon diffusion continues toward the graphite nodules as the solubility of carbon in the

austenite decreases with temperature [8, 12]. This is followed by eutectoid transformation, where austenite is converted to pearlite and multiple carbon atoms diffuse into nodules. Finally, depending on the amount of carbon (CE), the metal base of ductile iron can be ferritic, pearlite or a mixture of ferrite and pearlite. Some assumptions of the solidification process can be found due to the research of the microstructure obtained by quenching of nodular cast iron melt at different temperatures [8, 9]. The importance of nodular cast iron utilization is the result of a wide range of different qualities. Quality can be varied by combining different chemical compositions, melt treatments (inoculation, nodulation, alloying), different cooling rates leading resulting in variations of shape, size and number of graphite nodules [10, 11].

Extensive research of the cast iron microstructure development led to the creation of different theories about the solidification process. The aim is to enable the creation of models for prediction of the solidification process and the mechanism of the graphite nodule growth. The following two models attempt to explain the solidification process of nodular cast iron with the eutectic composition (CE = 3.4 %). These models are called uninodular (Figure 3 a) [8, 13, 14] and multinodular (Figure 3 b) theory [8, 15]. Uninodular theory assumes that graphite clusters nucleate in the melt and are separately surrounded by round austenitic shells. In this case both phases grow by diffusing carbon from the liquid into graphite clusters until the end of solidification, creating nodules. However, this form of graphite growth is difficult to explain theoretically due to the time required for carbon diffusion through austenite, as well as because of the pressures that should occur in the austenite shell when the graphite nodule grows.

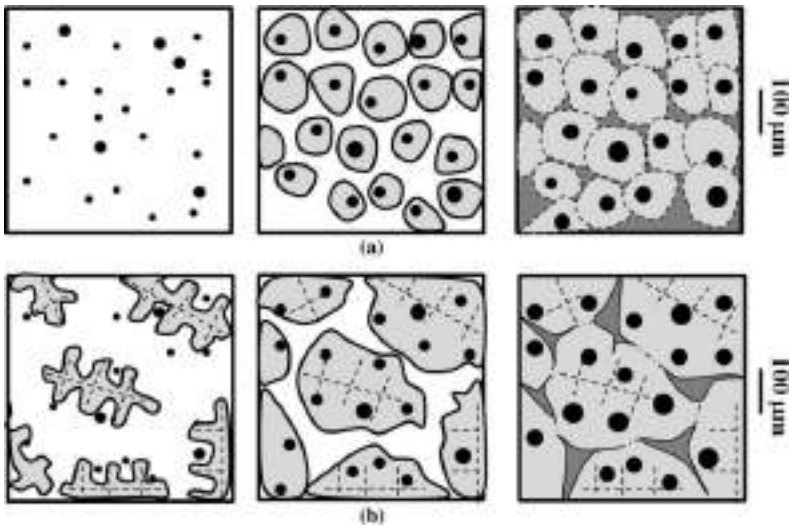

Figure 3. Microstructural evolution according to the a) uninodular
b) multinodular theories[14]

The multinodular theory [8, 14, 15] suggests that both phases nucleate independently in the melt. This theory is based on the assumption that austenite grows in dendritic form and with continued solidification, dendritic branches surround graphite nodules [15]. Uninodular and multinodular models are described in more detail in the literature [15, 16]. One of the most important mechanical properties of nodular iron castings is its ductility which is achieved by the nodular form of graphite present in the metal matrix [8].The growth of nodular graphite is limited due to the carbon diffusion through the austenite during the solidification process.

This limitation leads to the creation of preferential growth directions which obtain the shape similar to the rose lettice. The final shape of the graphite mainly depends on the rate at which the austenite wraps around the nodule. This rate depends on the undercooling of the casting, which is also affected by the amount of magnesium present in the melt [11, 12].
Theoretically three types of austenite encapsulation (surrounding the nodule with an austenitic envelope) can be distinguished: fast, slow, and non-existent encirclement [17]. If there is a rapid nodule encirclement, then the graphite has the same radii for carbon diffusion from all directions, resulting in round graphite. If wrapped slowly, the nodule will have an area of preferred growth, reducing the final nodularity. And finally, if there is no wrapping of the nodule with an austenitic sheath, the final geometry will correspond to laminar graphite [17, 18]. Although the multinodular theory is based on experimental findings of austenite dendrites in the microstructure of the eutectic composition alloy, more research has focused on simulating the solidification of nodular iron castings according to uninodular theory [12, 16, 19, 20] versus multinodular theory [8, 17, 18]. Existing solidification sequence prediction models assume that nucleation and growth of graphite will lead to the formation of a complete sphere, which is a theoretical and ideal behavior that does not occur in real production conditions [8, 13, 18].
The achievement of the required properties in nodular iron casting is depending on the mechanisms of solidification as well as inoculation, modification and degeneration within different solidification conditions [11, 12, 19]. Efforts are made in order to explain the nucleation process in iron-carbon alloys with graphite. A number of theories have been developed over the years. Nevertheless, significant questions remain unanswered. The reason lays in the vide variety of conditions, such as temperature range in which the nuclei are formed, the complexity of reactions that take place in the melt, the chemical composition of the nucleus, etc. It is difficult to give a clear and simple answer to the question of the course and mechanism of nucleation.

NUCLEATION THEORIES

The graphite nucleation mechanisms in the melt can generally be explained according to one of two theories. The first is the theory of homogeneous nucleation on a carbon-enriched cluster or on a particle of undissolved / residual graphite (single-phase nucleation) [12, 19]. The second is the theory of heterogeneous nucleation (single- or multi-phase nucleation). Even though the graphite-enriched particles are not considered homogeneous nuclei, they are taken into the account as homogeneous particles because they have the same chemical composition as the graphite phase growing on them. Both theories view residual graphite as the ideal nucleant for graphite formation during solidification [20, 21, 22]. The heterogeneous nucleation theory also points to the importance of the certain inclusions present in the melt as possible sites for heterogeneous nucleation of the graphite. Heterogeneous nucleation taking place in the real production conditions depends on the purity of the base metal, retention time, temperature and the melt processing. The inoculation is a melt treatment technique that manages the development of a microstructure which works by introducing particles into the melt. Those particles stimulate the accumulation of the suitable particles for the nucleation of graphite clusters. The commercial inoculants are based on a ferro-silicon master alloy containing the small amounts of elements such as Ca, Ba, Sr, Al, Zr, and rare earth elements [22, 23]. The significance of these elements is in their strong affinity for oxygen and sulfur in the melt.

When combined, those elements form complex oxides and sulfides which then act as the nucleation sites of graphite. Theories of heterogeneous nucleation developed in the recent history have mainly focused on non-metallic inclusions present in all commercial iron castings. The special requirements that the elements in the inoculant must meet in order to act as the potential nucleation sites in the melt are good crystallographic compatibility, small lattice irregularity, fine dispersion in the melt and high stability at elevated temperatures [19, 24]. Some of the most important theories of heterogeneous nucleation are mentioned below.

The gas bubble theory is based on the claim that tiny gas bubbles in the melt are ideal sites for the formation of nuclei on which the graphite particles can grow. According to this theory, the direction of growth of graphite is radially from the outside to the inside of the bubble. Gas bubble theory is in principle based on the presence of carbon monoxide in the melt. However, under industrial conditions of ductile iron melt production, strong deoxidants are added to remove dissolved oxygen. It is unlikely that the graphite cluster will expand in the direction of the internal volume of the gas bubble, because such growth would involve the diffusion of carbon through the graphite shell [19,25].

The graphite or carbon-rich cluster theory was introduced as a form of homogeneous nucleation. According to this theory, graphite grows from a small particle of crystalline graphite that is already present in the melt [16, 19, 26]. However, when the melt is treated with a ferro-silicon master alloy, Si promotes nucleation of the graphite. Since the dissolution time for the ferro-silicon master alloy in the melt is a few seconds, the theory assumes the formation and retention of particles in the melt. The condition for the retention of graphite clasters is that Ba or Sr are present in the melt in sufficient quantities to prevent the dissolving and to promote the formation of carbon clusters containing approximately 15 atoms. Therefore, in this theory, clusters of C_n or $(Fe_3C)_n$ exist in dynamic equilibrium in molten iron alloys with graphite and serve as sites for graphite nucleation [19]. Recent research has shown that in low-carbon gray cast irons, graphite nucleates at the austenite-liquid interface without the presence of foreign inclusions, which supports the theory of graphite nucleation on carbon-rich clusters [16, 26].

The silicon carbide theory is based on the presence of silicon carbide (SiC) crystals and an increased amount of graphite particles near the dissolving ferro-silicon master alloy has been observed [19, 27]. Because of this phenomenon, a theory of graphite nucleation and the mechanism of SiC particle fading has been created. The theory is based on the assumption that there is a local supersaturation of C and Si in the melt after dissolution of SiC, which gives the necessary driving force for homogeneous nucleation of graphite. The effect of fading is explained by the homogenization of C and Si in the melt through convection and diffusion [28]. However, the critical role of elements such as Ca, Sr, and Ba in the inoculant cannot be explained by this theory [19, 27, 28].

The silicate theory is based on the fact that the most inclusions in nodular cast iron are the primary or secondary product of magnesium melt processing [13, 19]. Hexagonal nuclei are composed of two-layer compounds of Mg or Ca sulfides composition surrounded by MgO, SiO_2 and 2MgO x SiO_2 with an epitaxial mechanism of graphite on oxide growth [22]. The hypothesis was supported by multiple studies that attempted to identify the chemical composition of the graphite nodule nucleus [19,.21, 22, 28].

The sulfide/oxide theory was developed when studies have suggested that sulfides, oxides, or nitrides, formed after inoculant addition, act as nucleation sites during graphite solidification [19, 28, 29]. The theory has been confirmed by research [30] which has indicated the importance of non-metallic inclusions. It was found that most graphite clusters in cast iron are associated with non-metallic inclusions, mainly magnesium and calcium sulfides. Graphite nucleation clusters for lamellar and spheroidal graphite are composed of complex oxides and sulfides. The spacing within the lattice for oxy-sulfide particles coincides with the spacing of the graphite lattice thus providing a suitable substrate for graphite growth [20, 28, 30].

GRAPHITE GROWTH

Favorable technological properties and relatively low production costs result in the increasingly frequent use of nodular cast iron in many industries [3, 5]. The properties of nodular cast iron are mainly influenced by cooling rate of products, addition of alloying elements, different casting technology application and cooling grate which can affect the shape of graphite and the formation of metal matrix [8, 10, 11]. Solidification process and the mechanism of graphite growth in nodular cast iron have not been fully understood despite a number of studies [12, 14, 18, 25]. Graphite crystallizes from the melt in different forms depending on the chemical composition of the melt and the solidification conditions (cooling rate). Basic morphologies of graphite present in iron cast alloys are: lamellar (LG), vermicular (CG) and nodular (SG) graphite. This transition is also based on changing of the growth direction of the graphite aggregate from the a- to c-direction, or from c- to a-direction. Chunky graphite is a highly branched, interconnected form of graphite, an it is considered degenerated form of spheroidal graphite [13, 22, 25]. The transition from lamellar to vermicular and from vermicular to nodular can be achieved by adding small amounts of Mg, Ce or lanthanide to the low-sulfur iron melt. This means that the process can be carried out in the opposite direction by adding sulfur or losing magnesium by evaporation and/or oxidation. The transition from vermicular to lacquered graphite is subject to the influence of certain elements used for production on melt processing [13, 25, 32]. Graphite forming in the shape of nodules attributes to high value of elongation and yield strength, good castability, machinability and toughness [22, 31].The study has been done on the problem of trace elements and their influence on the development and shape of graphite nodules [25, 32]. It was discovered that magnesium and rare earth elements stabilize the precipitation of oxides and sulfides by stimulating the growth of graphite nodules as shown in Figure 4.

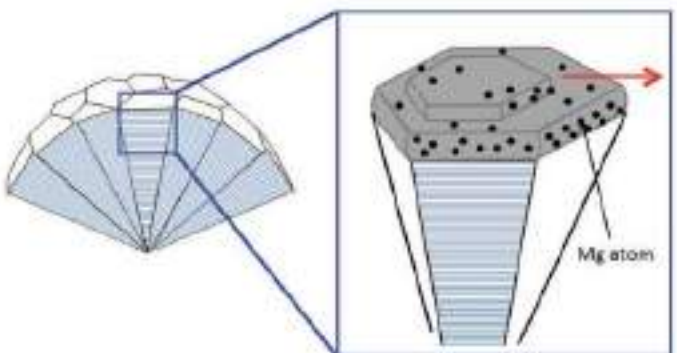

Figure 4. Schematic of spheroidal graphite growth [33]

Of all the elements present (except carbon) in nodular cast iron, silicon has the greatest influence on the carbon content in eutectic [24]. Today there are several nodular cast iron production processes. The base for all production processes is the modification of graphite form in the primary melt by modifiers (ferro-silicon master alloys) to obtain the characteristic spheroidal form of graphite [22, 27]. The commonly used modifiers are magnesium-based alloys that have a high affinity toward oxygen and sulfur in reference to the sulfide/oxide theory [19, 28]. This leads to the formation of stable oxides and sulfides which stimulate the growth of graphite nodules as shown in Figure 4 [32].

Alloying elements and cooling rate are the main factors influencing the formation of ferrite and pearlite as metal matrix [8]. Elements that promote the formation of pearlite are copper, chromium and manganese, but they also lower the temperature of eutectoid reaction and the rate of ferrite formation [19, 20]. With a proper content of elements that promote the formation of pearlite, nucleation will be possible at the boundaries of austenitic grains and at the interface of ferrite / austenite of graphite / austenite [21, 22].

Research on samples from thin-walled castings revealed that during high-temperature phase transformation in the solid state, nodules in the cast material have a dual microstructure [3, 23, 34]. In this case, graphite nodules consist of inner zone with disoriented graphite and the outer zone where the crystallization takes place properly. The disoriented zone is going to occur due to mechanical shrinkage deformation during solidification in a metastable system [34].

It is generally accepted that spheroidal graphite growth can be affected by surface-active impurities adsorbing on the unsaturated edges of graphite platelet leading to the change of preferred growth direction thus creating degenerated graphite nodules [13, 25, 35].

The sequence of solidification and the subsequent sequence of phase transformations in the solid state significantly affect the microstructure and mechanical properties of iron castings [19, 36]. It has been found that eutectoid transformation is a diffusion-controlled process and can be divided into two phases: ferrite formation (stable phase) and pearlite formation (metastable phase) from austenite [22, 36]. In the case of a low cooling rate, after solidification of the equilibrium phase there will be austenite and nodular graphite. The temperature decrease leads to the graphite nodules continuous growth due to carbon rejection by austenite [20, 36].

A review of the literature shows that the graphite growth and the course of solidification have significant impact on the microstructural and mechanical properties of nodular cast iron. However, the formation mechanism and thus the technology for degeneration prevention is still unknown.

CONCLUSIONS

The nucleation process and the mechanism of graphite growth in nodular cast iron have not been fully determined despite a large amount of research on the subject. Different experiments have led to the creation of theories about the solidification process and it is generally known that the sulfide/ oxide theory has been confirmed by research. It was found that most graphite clusters in cast iron are associated with non-metallic inclusions, mainly magnesium and calcium sulfides and that graphite nucleation clusters for lamellar and

spheroidal graphite are composed of complex oxides and sulfides. It is still uncertain what causes the changing of the growth direction of the graphite aggregate from the a- to c-direction, or from c- to a-direction. This change of growth direction causes graphite form degeneration which negatively affect mechanical properties of the cast. The aim of the future research should be the creation of models for prediction of the solidification process and the growth of graphite nodules in order to define optimal parameters for the nodular cast iron production process and help to achieve the desired properties of the casting.

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