Int. J. Mech. Eng. & Rob. Res. 2014

International Journal of Mechanical Engineering and Robotics Research

ISSN 2278 – 0149 www.ijmerr.com
Special Issue, Vol. 1, No. 1, January 2014
National Conference on "Recent Advances in Mechanical Engineering" RAME – 2014

Research Paper

GRAIN REFINEMENT OF CAST ALLOYS: A REVIEW

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The major disadvantage of coarse grain structure in castings is that it may result in a variety of surface defects and cracking. Non-uniformity of grain size also reduces the fatigue performance, yield strength and tensile elongation to fracture. A review made in the present study highlights the important methods of grain refinement for ferrous casting alloys. It has been shown that the processes used for the grain refinement of ferrous alloys require different types of techniques for the ferrous alloys with different carbon weight percentage. Techniques used for the refinement of ferrous alloys are equal channel angular pressing (ECAP), transformational grain refinement (TGR), current pulsing technique, conventional graphitising inoculant technique. It has been shown that grain refinement by using different techniques results in fracture toughness and high strength combination, improvement in low cycle, fatigue life, uniformity of properties, better feeding to eliminate shrinkage porosity and improved ability to achieve a uniform anodized surface. It has also been shown that grain refinement does not change the phase constitution, freezing characteristics and structural stability of the alloy and the stress rupture life of fine grained alloy sample is much better than that of conventional one. It has also been shown that there exists a relationship between the amount of grain refinement and the mechanical properties, which also depend on the refining method employed.

Keywords: Cast alloys, Grain refinement, Grain structure, Mechanical properties

INTRODUCTION

Casting is one of the oldest methods of manufacturing parts. In casting molten metal is poured into a mould cavity where, upon solidification, metal gets the shape of cavity. Solidification of any molten metal involves nucleation and growth. Nucleation appears in the molten metal in the form of tiny solid particles, called as nuclei, when phase transformation in the metal occurs. These nuclei are from by the deposition of atoms and grow into the form of crystal and these crystal results in the completely solidified grains. Nucleation is of two type- homogeneous nucleation and heterogeneous nucleation. Homogeneous nucleation does not need any

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foreign particle while heterogeneous nucleation needs foreign particle such as sand, dirt and impurities. Heterogeneous nucleation takes places at the surface and these grains are called as equiaxed grains while inside due to absence of any foreign particle homogeneous nucleation takes place and these grains are called as columnar grains and these columnar grains are not good for electromagnetic properties. These columnar grains grow rapidly and called as dendrites. These dendrites are undesirable in the casting so grain refinement is required (Khanna, 2012). Grain refinement is a technique used to improve the mechanical properties of the materials by decreasing their grain size. This is also called as inoculation. In this process the refining agents such as Al-5Ti-1B, are added to the material to work as nucleant. The major disadvantage of coarse grain structure is that it may result in a variety of surface defects in alloys used in rolling. Non uniformity of grain size also reduces the fatigue performance, yield strength and tensile elongation to fracture (Yuhu Xiang *et al.,* 2001). The main advantage of fine grain structure over conventional cast structure is fracture toughness and high strength combination, improvement in low cycle, fatigue life, uniformity of properties, comprehensive properties, compaction of fine powders, formation of a fine powders and randomly oriented equiaxed grain structure during solidification, crystallization of amorphous alloys, distribution of second phase and microporosity on fine scale, better feeding to eliminate shrinkage porosity, improved ability to achieve a uniform anodized surface (Yuhu Xiang *et al.,* 2001).

Refinement of cast structure can be produced by increasing the number of nucleating seeds, a large number of crystals are formed, impinge on each other and prevent each other from further growth. Grain refinement can be divided into three basic methods: mechanical, chemical and thermal. These methods involve: agitation of the melt during freezing, nucleation promoting and growth hindering additions and rapid cooling (Lin Liu *et al.,* 1998).

Reduction in grain size can be achieved in three different stages: during solidification of the molten metal, thermo-mechanical treatments involving recovery and recrystallization of the deformed material and severe plastic deformation. Grain refinement can effectively reduce hot tearing susceptibility during direct chill billet casting, ensures a uniform fine-grained recrystallized microstructure Lin Liu *et al.,* 2005).

GRAIN REFINEMENT OF FERROUS ALLOYS

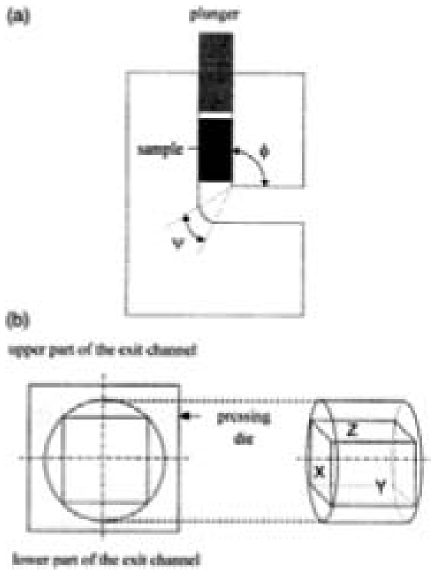
Ferrous alloys are that contain iron as the base metal. Basically the iron comes from the mines or rocks in the form of ores and separated from the ores. The members in the iron family are classified on the basis of carbon content present. In pig iron carbon content is 3.5-4.5%, wrought iron contains .05-.2% carbon, and cast iron contains 2.1-4% carbon while in steel 2.1% carbon is present. Grain refinement of the ferrous alloys can be done by the following processes (William, 1996).

Grain Refinement of Plain Carbon Steel

Plain carbon steel is an alloy of iron and carbon where the amount of carbon ranges from, 0.015% to 2 %. The material has great strength and hardness which are essential for motor bodies, appliances, apparatus, ships, containers, and the structures of buildings (William, 1996).

SHIN *et al.* (2001) has done the refinement of plain carbon steel during equal channel angular channel process (EACP), is one of the effective method of making of Ultrane-grained (UFG) materials (Dong *et al.,* 2001). UFG materials with submicro- meter-order grain size exhibit excellent mechanical properties compared with conventional ne-grained materials as well as coarse-grained materials (Segal *et al.*, 1981, Saito *et al.*, 1999, Valiev *et al.,* 2000, Liu *et al.,* 1998, Richert *et al.,* 1999). ECAP was carried on cylindrical specimen of f 18 mm ×130mm upto 8 passes with temperature of 673K, shown in Figure 1 (Dong Hyuk Shin *et al*., 2001).

Figure 1: (a) Schematic Diagram of ECAP Die (b) Diagram of the Sectioning the Pressed Sample for Optical Observation (Shin *et al.*, 2001)



The inner contact angle (ω) and arc of curvature (y) at the outer point of contact between channels of die, were 90º and 20º respectively and the diameter of the entrance channel, f18 mm was slightly greater than that of the exit channel f17.4mm because of the ease of the consequent passes. During ECAP, sample was rotated 180 º around its longitudinal axis between the passes. The pattern of the die in the shape of C was producing higher effective strain which was imposed on the same plane at every other passage. The route C was able to restore the shape of the original segment at each even number of passes and a nearly equiaxed ultrafine grain structure was obtained.

The microstructure was observed by slicing the sample into normal (X plane) or parallel(Y plane) to the longitudinal axis, as shown in Figure1(b). A 3% nital solution was used to etch the samples for the optical examination. TEM images and (SAD) selected-area diffraction pattern were obtained and examined. The optical microstructure, viewed on the Y-plane, shown in Figure 2. It was seen that the initial equiaxed grains, with grain size~30ìm, were elongated along the direction of 30º inclined to the longitudinal axis by the first pass. The second pass resulted in smaller and more irregular equiaxed grains with dim grain boundary but further passes brought morphological changes with an odd number of passes, the grains were severely elongated along the direction of 30º inclined to the longitudinal axis and grains were restored to the equiaxed shape with the consequent number of passes.

Figure 2: Optical Micrographs of as-Received and as-Pressed Low-Carbon steel: (a) as-Received (b) After One Pass (c) After Two Passes (d) After Four Passes (Shin *et al.*, 2001)

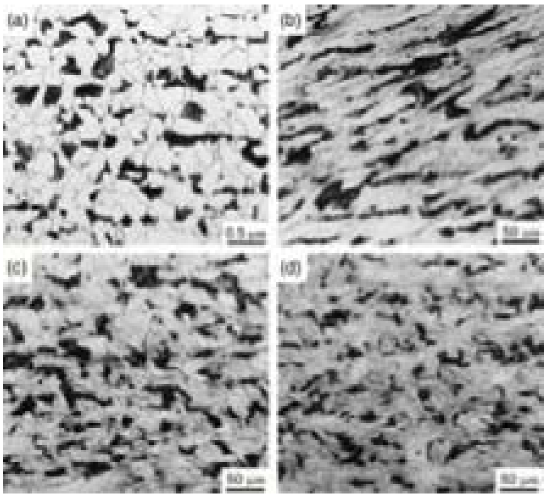
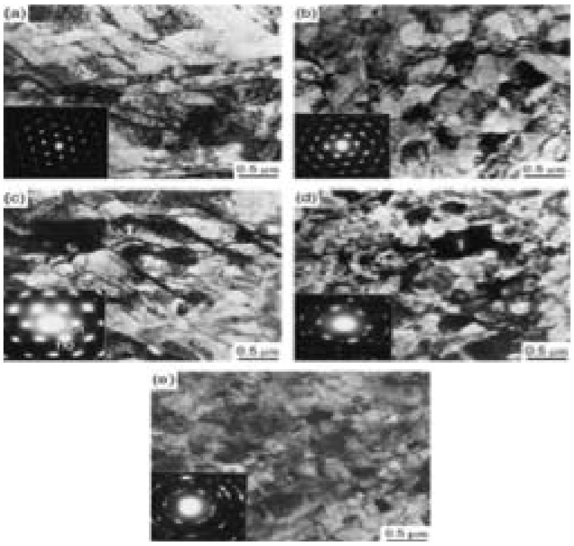


Figure 3(a) shows TEM micrographs and corresponding SAD pattern after the first pass, the ferrite microstructure (bright contrast) was seen with parallel bands of elongated grains having a width of ~0.3 µm. Figure 3(b) shows the pattern of second pass with grain size ~0.5 µm which was slightly larger than that of first pass. From SAD pattern it was noted that in this area the micro orientation between sub grains was increased compared with that in the sample with a single pass. Figure 3(c) presents the TEM morphology taken from a different region of the sample in Figure 3(b). The portion of ultrafine equiaxed grains was large though but some sub grain bands were still remained which shown that two passes of the ECAP were not enough to produce a considerable equiaxed structure. The microstructure after four and eight passes was shown in Figure 3(d) and Figure 3(e) with an average grain size of 0.2~0.3µm in both sample. It was seen that as number of passes pressing increased, the portion of high-angle grain boundary was also increased and resulted in the form of ultrafine grain structure (Dong, 2001).

Figure 3: TEM Micrographs Of The As-pressed Low-carbon Steel: (a) After One Pass; (b) After Two Passes; (c) After Two Passes [A Different Region Of The Sample In (B)]; (d) After Four Passes; (e) After Eight Passes (Shin *et al.*, 2001)

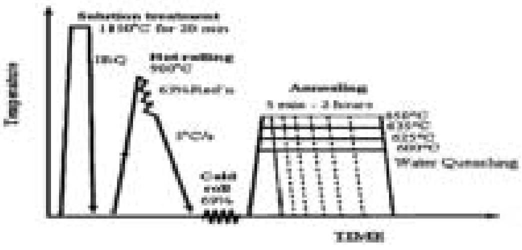


Grain Refinement of Alloy Steels

Any steel containing a notable quantity of some other metal alloyed with the iron, usually chromium, nickel, manganese, tungsten, or vanadium is called as alloy steel. Alloy steels are of two type, low alloy steel, medium alloy steel and high alloy steel. Low alloy steel contains carbon content to the range of 0.10 to 0.30 wt% which increases the weldability and formability of the steel while maintaining the strength. The medium carbon steels have carbon concentrations between about 0.25 to 0.60 wt% and used for producing railway wheels and tracks, gears, crankshafts and other machine parts. High carbon steel contains carbon content to the range of 0.60 to 1.4 wt% and are the hardest, strongest, and yet least ductile of the carbon steels. These are used to make tools and dies for forming and shaping materials (William, 1996).

Steels produced with more refined ferrite microstructure, improves the tensile properties of steel as compared conventionally produced by controlled rolling. Hodgson et al. first introduced the transformational grain refinement (TGR) processing steel strip to obtain a ferrite grain size of ~1µm in a substantial fraction of its volume (Hodgson, *et al.,* 1999, Hurley and Hodgson, 2001, Hurley *et al.,* 2000). Ahmad *et al*. (2005) has done the refinement of alloy steel with TGR processing. The vanadium microalloyed steel, provided in the form of hot- rolled slabs was used as a specimen. The specimen were hot rolled at 900ºC to reductions of 64% with the thickness of 3 mm and then cold rolled to a reduction of about 70%, with a final thickness of ~1mm as shown in Figure 4.

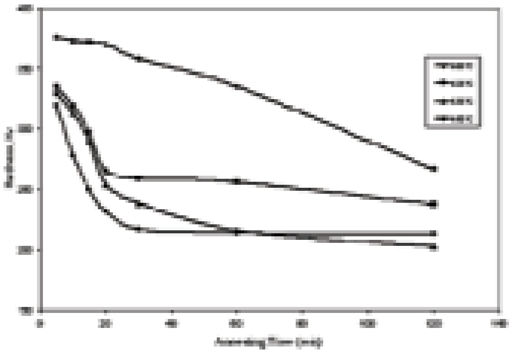
Figure 4: Schematic Representation of Rolling and Annealing Schedule (Ahmad *et al*., 2005)



After the grinding and polishing, the specimens were etched in 2% nital solution and Marshel's reagents to revel the ferrite grain boundaries. Hardness was measured in Vickers scale with 1kg load and electron backscattered diffraction (EBD) technique was used for the measurement of high-angle grain boundaries (HAGB) and low angle grain boundaries (LAGB). TEM was used for the vanadium precipitation study.

Ahmad *et al.* (2005) had done annealing after cold rolling and it promoted the grain refinement but also had an adverse effect on the hardness as a result of recovery. The annealing response to hardness is shown in Figure 5. The figure has shown that hardness dropped smoothly but more rapidly at a higher temperature (Ahmad *et al.,* 2006).

Figure 5: Dependence of Hardness on the Annealing Time and Temperature (Ahmad *et al*., 2005)



Priestner and Ibraheem (2000) in a similar study claimed that hardness drop was caused by the abnormal grain growth but Ahmad *et al.* had used V carbides that precipitated in the early stage of annealing and pinned the recrystallized grains, thus retarding abnormal grain growth (Priestner and Ibraheem, 2000). Figure 6 shows the TEM micrographs of V carbides taken on the thin film carbon replica, trapped on the copper mesh. The results of chemical analysis of these precipitates are shown in Figure 7. The precipitates were mostly very fine but some coarsening was also observed.

Figure 6: TEM images of V Precipitates: (a) Fine Dispersion (b) Some Coarser Precipitate (Ahmad *et al*., 2005)

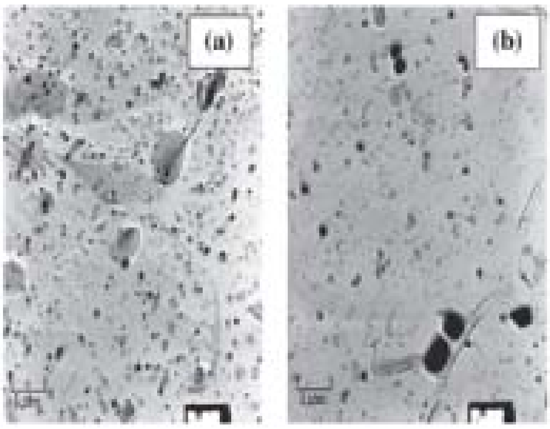


Figure 7: TEM Analysis of the V Precipitates Taken from Extraction Replica (Ahmad *et al*., 2005)

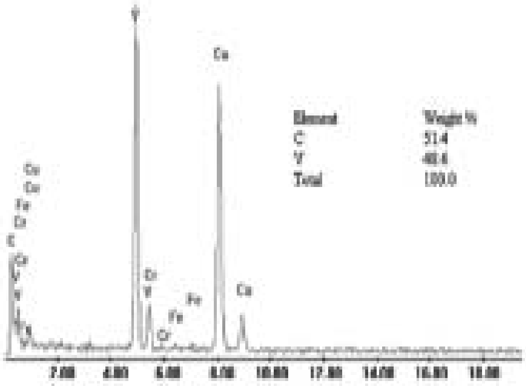
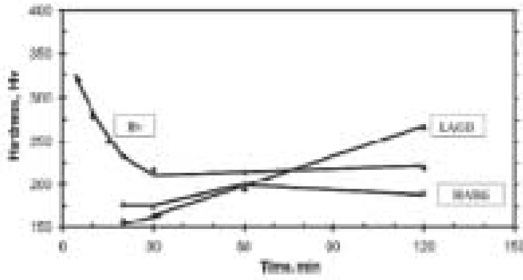


Figure 8 illustrates the changes in the HAGB and LAGB. The annealing process was started with LAGB and HAGB spacing, but the former increments with time were caused by the movement of dislocations, resulted in the merging of LAGB. The HAGB were not increased much by increasing the annealing temperature due to the V carbides and their pinning effect retarded the grain growth. The main problem occurred while using ultragrain refinement was the poor toughness but Ahmad et al. had prepared a mixed microstructure with HAGB an LAGB, with minimum coarsening even after 2 h of annealing, was rectifying the problem because vanadium carbonitrites pinned the grain growth more effectively than Nb (Ahmad *et al.*, 2005)

Figure 8: Dependence of Hardness and Boundary Spacing on Annealing Time at 600°C (Ahmad *et al*., 2005)



Grain Refinement of Stainless Steels

These are highly corrosion resistant in various atmospheres. The predominant alloying element is chromium with at least 11 wt%. Nickel and molybdenum present in stainless steel enhances the corrosion resistant properties. It is mainly used for making of valves, chemical and food processing equipments, rifles barrels, jet engine parts etc. Stainless steels are of three types- martensitic, ferritic or austenitic. Martensitic (0.15 C, 12.5 Cr, and 1.0 Mn) and ferritic stainless steel (0.06C, 17.0 Cr, 0.4 Mn, 0.40 Si, and 0.026 P) are magnetic while austenitic stainless steel (0.08 C, 19 Cr, 9 Ni, and 2.0 Mn) is not (Wiliam, 1996).

Reddy and Mohandas (2001) found that ferritic stainless steels can be used instead of austenitic stainless steels because of low cost and also have greater resistance to stress concentration cracking. Ferritic stainless steels are free from nickel and can be used where nickel undergoes leaching effect. Reddy and Mohandas (2001) concluded that one of the major problem faced in welding ferritic stainless steel was grain coarsening in weldments. Refinement of grain size in the welds was not possible during post weld solidification due to the absence of any phase transformation. It was also noted that grain refinement of welds can be done by electromagnetic stirring, current pulsing and liquid metal chilling. Reddy and Mohandas (2001) had done the grain refinement of ferritic welds by current pulsing. Ferritic stainless steel AISI 430 was taken as the specimen with following composition given in Table 1.

Table 2 shows the welding parameters used for the work. The welds were characterized by optical microscope examination and all-weld tensile property evaluation, which is shown in Figure 9.

It was also seen that because of AC current, current pulse technique was refining grains finer and equiaxed than DC current pulses welds. Figure 11 shows the fusion grain structure of DC and AC current pulsed welds.

From the Table 3 it was seen that AC pulsed welds were stronger and had better ductility than DC welds.

The refinement of weld zone grain size in pulsed AC current technique was done by the

Table 1: Chemical Composition AISI 430 of Ferritic Stainless Steel (Reddy and Mohandas, 2001)

C	Mn	Si	S	P	Cr
0.06	0.4	0.40	<0.010	0.026	17

Table 2: Welding Parameters (Reddy and Mohandas, 2001)

For unpulsed DC and AC

Arc current (amps): 180

Arc voltage(volts): 20

Travel speed (mm/s): 5

For pulsed DC and AC

Pulse current (amps) (Ip): 300

Background current (amps): 30

Pulse on time (%):20

Pulse frequency (Hz): 6

Arc voltage (volts):18-20

Travel speed (mm/s): 2.7

Figure 9: Shows DC and AC Continuous Current Welds With Columnar Grains, AC Current Welds Were Having Finer Grain Size and Presence of Equiaxed Grains

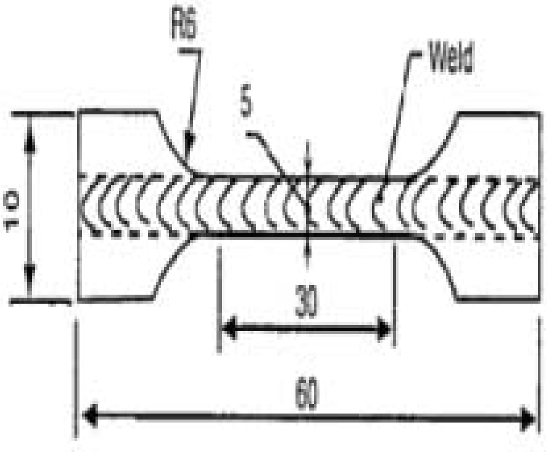


Figure 10: Fusion zone grain structure (a, b) Direct Current Continuous Weld (c, d) Alternating Current Continuous Weld (Reddy and Mohandas, 2001)

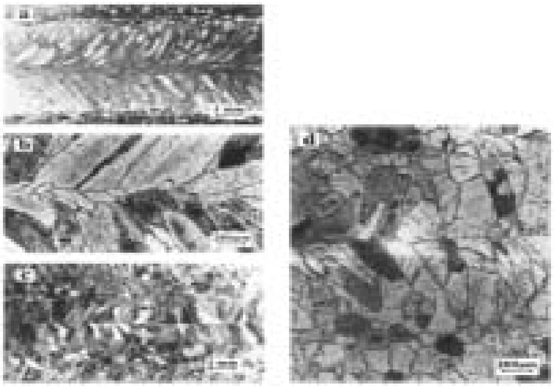
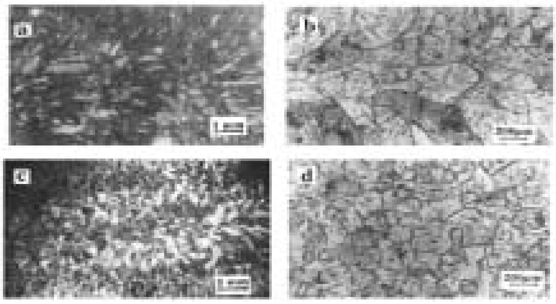


Figure 11: Fusion Zone Grain Structure (a, b) Direct Current Pulsed Weld (c, d) Alternating Current Pulsed Weld (Reddy and Mohandas, 2001)



introduction of convection in the weld pool leading to dendrite fragmentation and grain detachment from the fusion boundary. It was seen that greater agitation in the weld pool was due to the pulsed AC (Reddy and Mohandas, 2001). Yamamoto *et al*. (1993) showed that the molten pool vibrations, presented in modulated DC pulsed gas metal arc welding, were negligible in unmodulated DC pulsed. It was also seen that Lorentz forces were more effective in AC welding. It was resulted in more effective melt pool convection which was increasing grain detachment (Yamamoto *et al*., 1993).

Grain Refinement of Cast Iron

Cast iron is a ferrous alloy with carbon content above 2.14 wt%. Most cast irons contain between 3.0-4.5 wt% C and other alloying elements. The most common cast iron types are gray (C 2.5-4 wt%, Si 1.0-3.0 wt %), nodular (C 1.8-3.6 wt%, Si 0.5-1.9 wt %), white (C 1.8-3.6 wt%, Si 0.5-1.9 wt %), and malleable (C 2.16-2.90, Si 0.90-1.90 wt %) cast iron. Cast iron is used to make small cylinder blocks, cylinder heads, pistons, clutch pipes, liners, pinions, gears, rollers etc (William, 1996).

Inoculation is a metallurgical treatment used to improve the mechanical properties of alloys. Treatment is done by addition of small amount of inoculant that is capable of increasing the number of active nuclei which results in following changes:

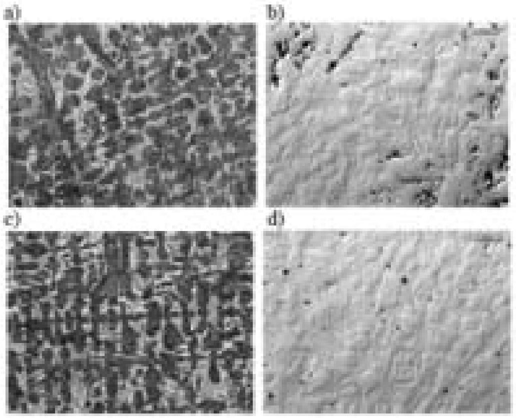
- Number of eutectic grains (increase),
- Undercooling degree during eutectic crystallization (decrease),
- Character of metallic matrix (pearlitic matrix with varied degree of dispersion) [20-28].

Kopycinski and Guzik (2008) had done the inoculation of low sulphur cast iron. Test melts were prepared by melting the charge, overheating to 1490º and melts were held at that temperature for about 100 seconds, and then additional inoculants (Fe powder and disintegrated steel scrap) were added to the molten alloy at the temperature of 1460ºC and SB5 master inoculant was mixed at 1410ºC. Nine samples were prepared with the composition given Table 4 and Figure 12 shows the microstructures of cast iron from melts Nos.1 and 2. Figure 12(a, b) was showing that, besides the dendrites of reacted austenite, the preponderate constituent was

Table 3: Longitudinal (all-weld) Tensile Properties (Reddy and Mohandas, 2001)

Type of Weld	Ultimate tensile strength (MPa)		0.2% Yield strength (MPa)		Elongation (%)	
	Continuous Current	Pulsed Current	Continuous Current	Pulsed Current	Continuous Current	Pulsed Current
Direct current	430	485	325	357	3	5
Alternative current	590	695	385	410	5.8	9

Figure 12: Microstructures of Cast Iron from Melts No. 1 - (a, b) and No. 2 - (c, d) (Kopycinski and Guzik, 2008)



cementite eutectic, due to the presence of Fe powder. The inoculation results in refinement of the white cast iron structure, as revealed by microstructures examined in Nomarsky contrast Figure 12 (b, d)

The inoculation carried out with SB5 master inoculant is shown in Figure 13. It is seen from the Figure 13 (a, d) that addition of master inoculant as well as Fe powder and disintegrated steel did not change the graphite precipitates but slightly increased the tensile strength UTS shown in Table 4 and in Figure 14 (cast iron with 0.08%) .

It was seen that addition of Fe power, indirectly affected the number of graphite eutectic grains, increased it slightly as shown in Figure 15. This effect was due to an increased number

of the austenitic grains and smaller dendrite arm spacing, leaving less free space for the crystallization of graphite grains. From the results of the investigations compared in Table 4 and Figure 15 it followed that sulphur drop in cast iron after inoculation considerably reduced the tensile strength UTS (melts Nos.

Figure 13: Graphite Distribution in Iron Casting Microstructure Produced from Melts No. 3 - (a, b), No. 4 - (c, d) and No. 5 - (e, f) (Kopycinski and Guzik, 2008)

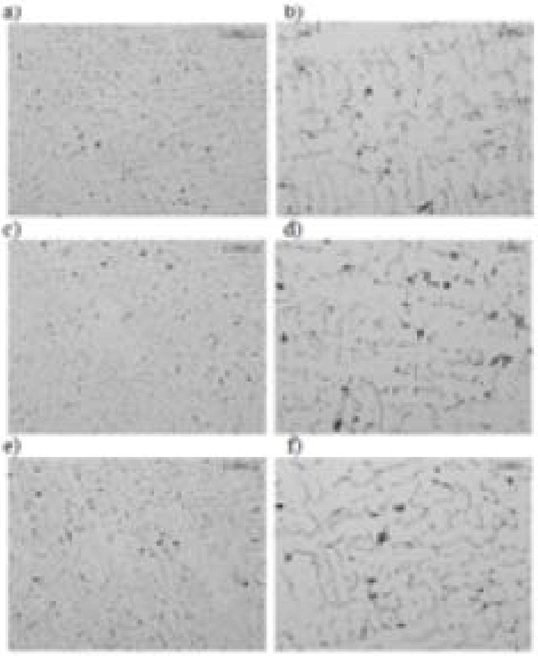


Figure 14: Sulphur Content and Inoculation Type vs. Tensile Strength UTS of Cast Iron (Kopycinski and Guzik, 2008)

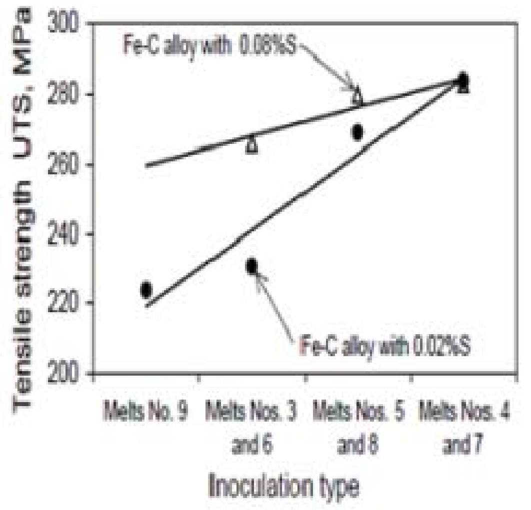


Figure 15: Number of Graphite Eutectic Grains in the Structure of Cast Iron from Melts No. 3 - (a) and No. 4 - (b) (Kopycinski and Guzik, 2008)

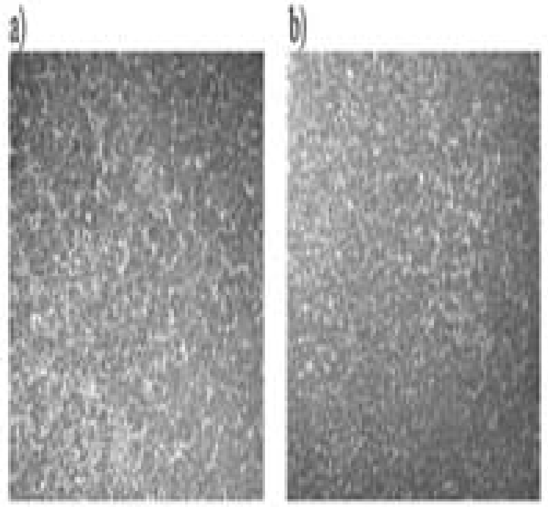


Table 4 Melt specification. (Kopycinski and Guzik, 2008)

Melt No.	Sulphur Content Wt%	Description of Metallurgical Treatment	Tensile strength UTS, MPa			
			UTS1	UTS2	UTS3	Mean
1.	0.08	Base cast iron,plain	-	-	-	**-**
2.	0.08	Base cast iron with addition of Fe powder, Without master inoculants SB5	-	-	-	**-**
3.	0.08	Inoculation with SB5	251	269	279	**266**
4.	0.08	Adding Fe powder,inoculation with SB5	271	281	288	**280**
5.	0.08	Adding disintegrated steel scrap, inoculation with SB5	272	285	292	**283**
6.	0.02	Inoculation with SB5	224	231	238	**231**
7.	0.02	Adding Fe powder,inoculation with SB5	278	282	292	**284**
8.	0.02	Adding disintegrated steel scrap, inoculation with SB5	264	269	273	**269**
9.	0.02	Physical mixture (SB5 inoculant and disintegrated steel scrap)	218	223	231	**224**

3 and 6 in Table 4). Introduced additional inoculants, i.e. Fe powder or disintegrated steel scrap, to molten alloy shortly before pouring of mould improved the mechanical properties (melts Nos. 7 and 8 in Table 4 and Figure 15).

Figure 16 shows the microstructure of cast iron in case of precipitates of graphite were of an interdendritic character and type E which meant the small dendrite arms spacing, smaller than the interlamellar spacing in graphite. Figure 17 shows Microstructures of the examined cast iron proved the existence of pearlitic matrix in the structure of cast iron after an additional treatment, i.e. with Fe powder. Similar structure was obtained after an addition of the disintegrated steel scrap (Kopycinski and Guzik, 2008).

Figure 16: Graphite Distribution in Iron Casting Structure Produced from Melts No. 6 - (a, b), No. 7- (c, d) and No. 8 - (e, f) (Kopycinski and Guzik, 2008)

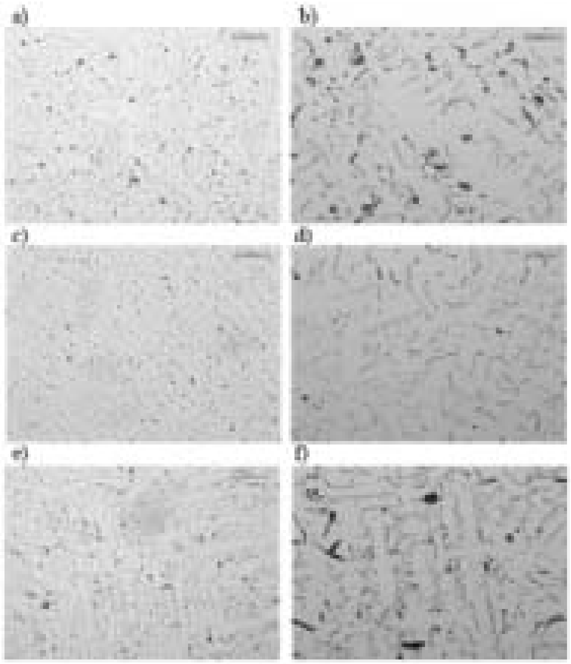
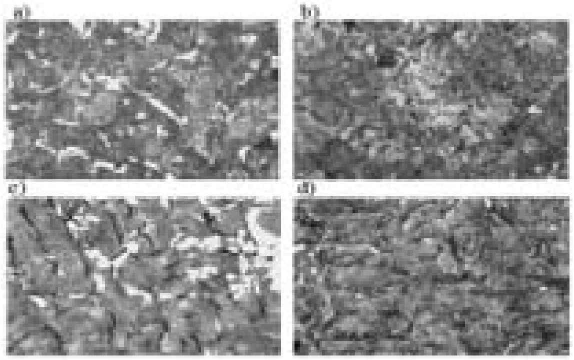


Figure 17: Matrix of Iron Casting Structure Produced from Melts No. 3 - (a), No. 4 - (b), No.6 - (c) and No. 7 - (d) (Kopycinski and Guzik 2008)



DISCUSSION

It was seen in the refinement of plain carbon steel with ECAP, when the sample was rotated 180° around its axis between passages, fine shear bands were formed for an odd number of passes, while even numbers of passes were dominant in restoration of the initial equiaxed grain shape. As the number of pressing passes increased, rotation of subgrains with the serrated boundaries was also increased. Subgrain rotation was more favourable than intragranular strain for accommodating a large amount of strain since serrated boundaries restrict dislocation movement causing intragranular strain. Refinement of alloys steels with TGR, refined grain size was from 50 to 2.8 μm. The V precipitates dissolved in the earlier process by annealing at 1150°C, would have precipitated out during heating to 900°C and slowed down the recovery during hot rolling. During annealing of cold-rolled specimens, enough stored energy was provided for recrystallization, but the process of abnormal grain growth was sufficiently slowed down due to pinning of V precipitates, resulted in effectively controlled the grain size.

Refinement of stainless steel welds was done by current pulses was showing that the AC current pulsed welds were stronger and more ductile than that of DC. Refined grain structure was easily achieved. Inoculation of low sulphur cast iron with SB5 master alloy was the number of crystallisation nuclei of the dendrites of primary austenite. The iron particles was acting as substrates for the nucleation of primary austenite Y_p due to a similar crystallographic behaviour of the regular face centered cubic lattice. The more numerous were the dendrites of primary austenite, the less free space was available in the interdendritic spaces for the formation of graphite eutectic grains, which made the structure more refined (more eutectic grains) and the mechanical properties higher.

CONCLUSION

It was seen from the previous studies that grain refinement is necessary for casting for its mechanical properties. It can be achieved by low the pouring temperature, by adding any refiner or by any technique. It was seen that there is a decrease in mechanical properties with large grain size.

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