# The effect of two-step solution heat treatment on the impact properties of Hadfield austenitic manganese steel

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**Abstract**. Hadfield austenitic manganese steel is made of iron with 1.0–1.4 percentage Carbon and 10–14 percentage Manganese. Hadfield steel was processed to solution heat treatment to eliminate the carbide (Fe,Mn)3C. This study aims to make a smaller grain size of Hadfield steel at once with solution heat treatment. It was expected to improve toughness of Hadfield steel. Solution heat treatment was carried out in stages by implementing pre-isothermal heating at a lower temperature with two variations at 600 degree C and 700 degree C before undergoing high austenitization heating. Pre-isothermal heat at above 450 degree C was promoting pearlite growth. Pearlite growth starting from austenite grain boundary then reformed new grain, which has a smaller size than prior austenite. Second, stage of austenitization heating then was performed at 980 degree C to transform new small grain pearlite to austenite. An agitated water quench was used to ensure a faster cooling rate to achieve 100 percentage austenite structure. Results demonstrate that sample that underwent a stepped heat treatment process at 600 degree C followed by austenitization at 980 degree C produced finer (smaller) austenite grains. That sample had the highest impact value of 329.1 J/Cm2 in comparison to other specimens.

## 1. Introduction

Hadfield austenitic manganese steel is made of iron with a composition of 1.0–1.4% Carbon (C) and 10–14% manganese (Mn), with a C : Mn ratio of 1:10 [1, 2]. The Mn alloy function as austenite phase stabilizers at room temperature [3, 4]. This type of steel was discovered in 1883 by Sir. Robert Hadfield (Sheffield, UK) [4]. Manganese steel is well-known in the steel industry because of its unique characteristics; it has exceptional toughness, high ductility, high work hardening ability, and excellent wear resistance. Because of the combination of these properties, manganese steel has been recognized as an advantageous engineering material [4]. The toughness properties are related to the presence of austenitic structures, which are formed after the material undergoes solution heat treatment [4-5]. Because austenitic matrix structures with a face-centered cubic (FCC) crystalline structure have more slip plane and direction than body-centred cubic (BCC) structures, they are capable of significant elongation; therefore, they have a high impact value [3, 4]. The excellent manganese steel wear-resistance properties are related to the material's ability to harden very rapidly upon plastic deformation. Previous studies have reported that the strain hardening in manganese steel was caused by the strain-induced transformation of $\gamma$ to $\alpha$ or $\varepsilon$-martensite [6]. Smith [7] reported that strain-induced transformations occur due to local decarburization or manganese segregation processes, which cause unstable austenitic conditions to be easily transformed into hard and wear-resistant martensite. Due to

its beneficial properties, manganese steel is widely used in the mining industry, for excavations, for oil drilling, in steelmaking, in railroads, for dredging, for naval-related equipment, for bucket wheel excavator parts, and for mineral mining equipment [8].

When producing manganese steel, two main processes need to be considered. The first process involves melting the raw material and achieving the right composition of C and Mn with a 1:10 ratio, according to the targeted composition. The second process entails implementing the appropriate heat treatment process to obtain a 100% austenite microstructure. The microstructure of as-cast manganese steel is austenite with the presence of carbide $(Fe,Mn)_3C$ and a small amount of pearlite. $(Fe,Mn)_3C$ is formed and grows at the grain boundary between the dendrites, resulting in brittle properties. Thus, it is necessary to apply the heat treatment process to as-cast manganese steel, particularly the solution heat treatment, to dissolve $(Fe,Mn)_3C$ into a single-phase austenite matrix to improve the toughness of the material [1]. Conventional heat treatment methods, known as solution heat treatments, are carried out to decompose the $(Fe,Mn)_3C$. The austenitizing temperature needs to range from 30°C to 50°C above the Acm line. It has been recommended that the temperatures range from 1010°C to 1120°C [4].

The researchers have studied improving the mechanical properties of metals in general. The improvement which has been carried out includes refining grains. In line with Hall-Petch's theory, where refining grains can increase strength [9]. The techniques of processing using a particular alloying element [7, 10], thermal or heat treatment [11], and mechanical treatment, which called severe plastic deformation (SPD) producing microstructure to UFG (ultra-fined grain) [12]. Smith [7] gave Vanadium to improve the yield strength, hardness and wear resistance qualities of manganese steel. But there was a decrease in the value of toughness. This low value can be understood because the addition of vanadium encourages the formation of carbides. One suggested the approach is to reduce the Carbon content so that it can reduce the formation of carbide volume fractions. Venturelli [10] perform the austenite grain refinement by Hafnium inoculation. The microstructural characterization result showed that the Hf-refined cast manganese steel was producing a grain size of 600 μm, whereas the non-refined material has 3000 μm. The result of the mechanical test showed that the austenite grain refinement promoted an increase in the toughness up to 88%. Martin [11] found that the application of isothermal heating at the manganese steel above 450°C will promote pearlite growth. The pearlite growth begins from the austenite grain boundary or can be grown intragranular. The pearlite growth builds the new grain and has a smaller size than prior austenite.

Based on the works of literature above, it can be deduced that the improvement of the mechanical properties of manganese steel can be reached by modification of the solution heat treatment. This research does not consider the option to use special alloying elements or SPD techniques. The critical parameters in the solution heat treatment process are austenitization temperature, holding time, and quenching rate. It was also found that the long heating process at high-temperature results in decarburization and loss of manganese elements and produces ε-martensite structure at the surface [3, 13], which contributes to the brittleness of the material. This process drives grain growth and to avoid loss of C and Mn elements and excessive growth of grains during the heating procesets. It is necessary to set appropriate austenitization temperature and holding time. In this research, the carbon content was reduced below 1.2 % to minimized the carbide formation [7]. Due to lower carbon content than the austenitization temperature can be set lower [3], and the heating time is set shorter for the effective heating process and to avoid excessive grain growth [13]. The heating process was conducted slowly to prevent internal cracks. Then, the quenching process is made fast-enough to minimize the reformation of $(Fe,Mn)_3C$ when quenching is carried out [14]. There were two steps involved in ensuring that the heating process reaches the center of the material – pre-isothermal heating and application of austenitizing temperature. Pre-isothermal heating is performed at a lower temperature before the austenitic temperature to reduce the overall holding time required at high austenitizing temperatures. This research was also utilizing the phenomenon of pearlite formation at isothermal heating above 450°C as stated by Martin [11]. The big austenite grain will be rebuilt by the new pearlite growth to be finer grain [11].

Therefore, this research intends to study the effect of step heat treatment temperature on the impact properties of manganese steel material. It is expected that with the use of step heat treatment methods, there will be an effective and efficient heating process to bring about the formation of 100% austenite structure and also to produce a finer grain to increase the impact value of the manganese steel. Impact value represents the toughness of the material. Finer grains provide more grain boundaries. The more grain boundaries gain, the more difficult for the dislocations to move and to result in higher strength [9]. The high-manganese steel specimen with small grain size, giving the improvement of the ductility because the formation of deformation-induced martensite is suppressed during tensile testing [15]. The high strength and ductility made the metal becomes tougher. Grain refinement is a really an advantageous concept in metallurgy because it is the only process that can create an increase of strength and toughness simultaneously on the metal.

## 2. Methodology

### 2.1. Material

The samples were made in a cylindrical form with a diameter of 20 mm according to Japan Industrial Standard JIS G 0307. These samples were cast through the use of a $CO_2$ process quartz sand molding. The melting process was carried out with the use of a medium frequency induction furnace of Inductotherm. The chemical composition of the material was checked by OES (Optical Emission Spectrometry) of ARLTM 3460 Thermo Fischer Scientific. The result of the chemical composition is as shown in Table 1 below.

**Tabel 1.** Chemical composition of manganese steel.

|            | C    | Si  | Mn    | P    | S     | Cr  |
|------------|------|-----|-------|------|-------|-----|
| OES Result | 1.11 | 0.4 | 13.25 | 0.05 | 0.017 | 0.9 |

The as-cast sample was prepared for metallographic examination with 3% nital etching. Observations were made with the use of a Scanning Electron Microscope (SEM) of Hitachi SU 3500, and the results of the observations are as shown in Figure 1. In the structure, carbides $(Fe, Mn)_3C$ which were to be removed by heat treatment methods, were observed at the grain boundaries.
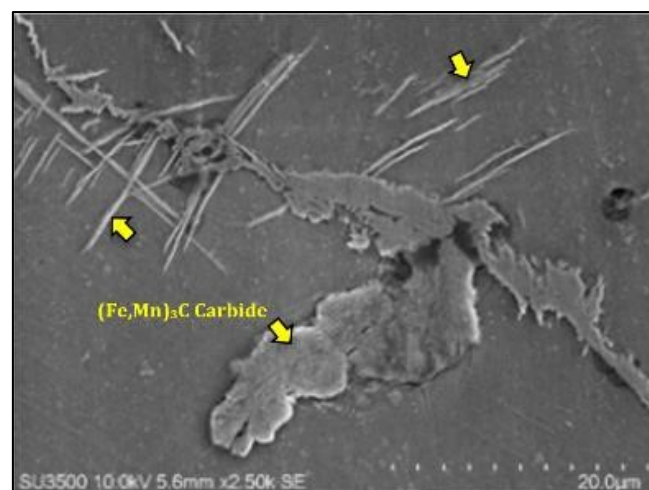


**Figure 1.** The microstructure of as-cast manganese steel.

The results of the EDS (Energy-dispersive X-ray spectroscopy) analysis are as shown in Figure 2A, B, and C. Figure 2A shows the observed area. Area 1 is the area of the austenite matrix, while area 2 is for the carbide. Figure 2B shows the EDS results for area 1 where the Mn level (10.76%) was found to

be smaller than the Mn level in area 2 (13.61%) (Figure 2C). Mn content in carbide is higher than the one in the austenite matrix, and this shows that the carbide formed is rich in Mn of $(Fe,Mn)_3C$.
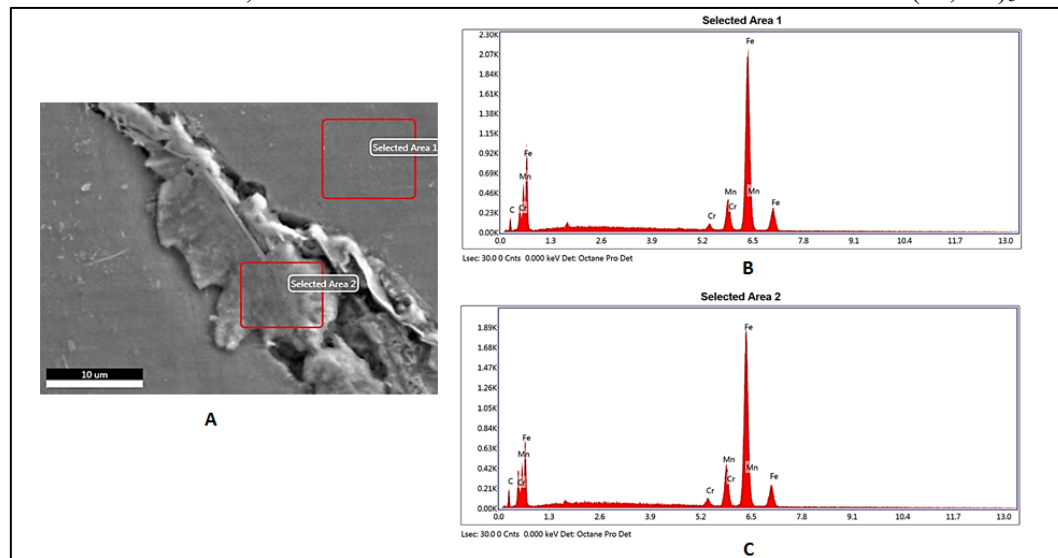


**Figure 2.** EDS observation of carbide and austenite matrix (A. EDS selected area of EDS observation, B. Selected area 1 result: C 7.55%; Cr 1.18%; Mn 10.76%; Fe 80.51%, C. Selected area 2 result: C 9.72%; Cr 1.48%; Mn 13.61%; Fe 75.19%).

## 2.2. Mechanical testing and metallographic examination

Hardness testing was conducted through the use of the Rockwell B Hardness method (HRB) according to JIS Z 2245 standard using Future-tech FR-1e hardness testing machine. Impact testing was done with the Hung Ta HT-8041A impact testing machine, using Charpy methods by the JIS Z 2242 standard. The V-notch type of test sample was applied with specifications, according to JIS Z 2202. A standard metallography examination was performed using an optical microscope and SEM to observe the microstructure formed in the cast material as well as after the heat treatment processes. The etching process made use of 3% nital, with 5 seconds of etching time for the as-cast sample and 30 seconds for samples that were given the heat treatment process.

## 2.3. Heat treatment

The principle that can be applied to obtain fine grains is to promote the growth of pearlite, which begins from the austenite grain boundary. Its growth can split the big austenite grain into a finer size. The pearlite growth can occur at a temperature range of 450°C–800°C [11]. The next critical parameter is the austenitization temperature. Determination of austenitization temperature is important in the solution heat-treatment process. At austenitization temperatures, all carbides will dissolve into the austenite matrix. The austenitization temperature is a function of Carbon content. The lower the carbon composition in the material to have a lower austenitization temperature limit. Referring to the pseudo-ternary diagram which was presented by Srivastava and Das [3]. The boundary of austenite area at 1.11% Carbon lay down at a temperature of 950°C so that austenitization temperature can be applied slightly above that temperature, i.e., 980°C.

To be able to do the two principles as stated above, then the heat treatment experiment was carried out through the use of the stepped heat treatment procedure. This procedure was done by carrying out a pre-isothermal heating process before the application of the austenitizing temperature. The variations in the heat treatment process applied to the samples are as shown in Table 2 below.

**Tabel 2.** Heat treatment variation.

| No | Heat Treatment | Sample Code |
|----|----------------|-------------|
| 1 | As-Cast | AC |
| 2 | Heat 1100°C 2H – Water Quench Agitated | HT 1100 |
| 3 | Preheat 700°C 3H – 1000°C 1.5 H – Water Quench Agitated | Step HT 700-1000 |
| 4 | Preheat 600°C 10 H – 980°C 2 H – Water Quench Agitated | Step HT 600-980 |
| 5 | Isothermal 600°C 10 H – Water Quench | Iso HT 600 |

The cooling process was carried out through the use of agitated water. The agitation was done to ensure that that the cooling rate was fast enough to avoid the reformation of carbide. The first step temperature was varied to determine its pearlite growth effect on the producing of finer grain. The austenitization temperature was various to know the effects it has on grain growth. The lower the austenitization temperature, result in the lower the grain growth rate. Therefore, it was expected to produce finer grains. Sample Iso HT 600 was performed to ensure the formation of pearlite at the range of temperature 450°C–800°C.

## 3. Results and discussion
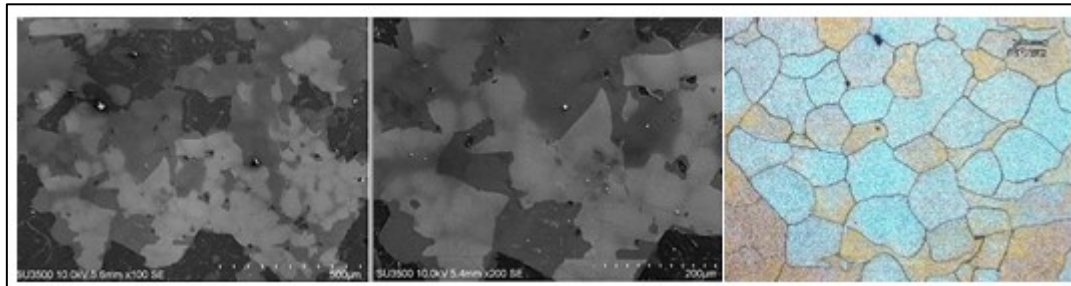
### 3.1. Microstructures
In comparison with the as-cast microstructure, all samples that were processed by heat treatment did not appear to have any carbide Figure 3,4 and 5. This condition can be explained that while the material was heated to austenitization temperature, all structure transforms into 100% austenite. Manganese steel with the content of C 1.11% has the austenite transformation temperature boundary starting at 950°C [3]. Above the temperature, the carbide $(Fe,Mn)_3C$ is dissolved into the austenite matrix to become a solid solution. The rapid cooling was executed preventing the carbide reformation [16]. The cooling process should also be ensured to pass the range temperature of carbide sensitization at 400-800°C fast enough. The slow cooling will stimulate a carbide formation. The quenching process of this research was using agitated water, through this method, the rate of cooling is maintained fast enough to prevent the carbide reformation.
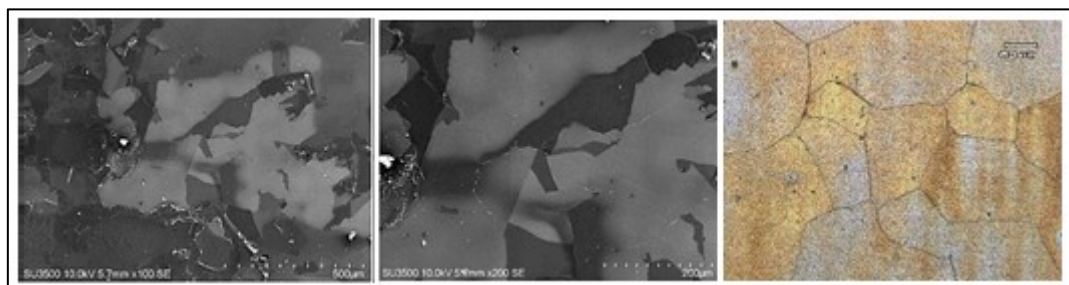


**Figure 3.** The microstructure of HT 1100.
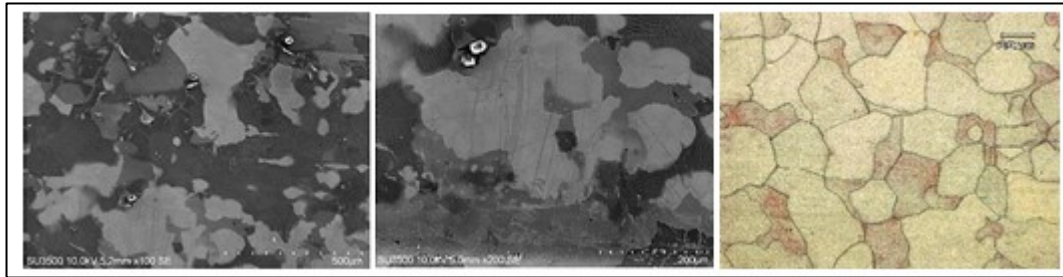
**Figure 4.** The microstructure of step HT 700-1000.



**Figure 5.** The microstructure of step HT 600-980.

Figure 3, 4 and 5 shows the microstructure of manganese steel samples that were processed using solution heat treatment, with and without pre-isothermal heating. The grain size for the step HT 600-980 sample is finer compared to other samples. The grain size was calculated using intercept methods. The grain size of sample HT 1100 is 151.27 micron, HT 700-1000 is 70.51 micron, and HT 600-980 is 68.20 micron. The smaller grain size provides the samples with a pre-isothermal heating process. It has approximately size of 45% lower compared to the grain size of conventional heat treatment. On manganese steel, the pearlitic reaction occurs above 450°C and begins on the grain boundaries [11]. The heating isothermally at 450°C and above will promote the growth of pearlite. This phenomenon can be illustrated by the evolution of the microstructure as shown in Figure 6. It illustrates the as-cast austenite ($\gamma$) grain, which has a big size. When the materials were heated and held isothermally at a temperature of 600°C and 700°C, then the pearlite growth started to begin at the grain boundary. The pearlitic structure reconstructs the new smaller grain. The heating continues to austenitization temperature, then the pearlite transforms to austenite, and the austenite grain remains as pearlite grain. To prove the pearlite formation on the manganese steel, then the experiment of sample Iso HT 600 was accomplished. The operation was done by heating the sample to 600°C and hold for 10 hours, then quench to water. The result as shown in Figure 7.
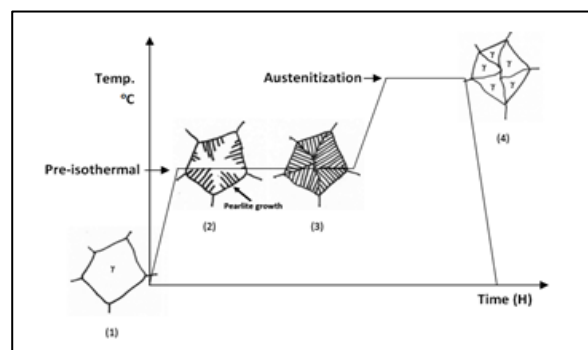


**Figure 6.** Evolution of microstructure during step heat treatment process.

Figure 7 shows the pearlite, which is grown from the grain boundary, and also found the intragranular pearlite formation. This result conforms as Martin [11] states that on the isothermal heating at the temperature above 450°C, can be found in the colony of intragranular pearlite.
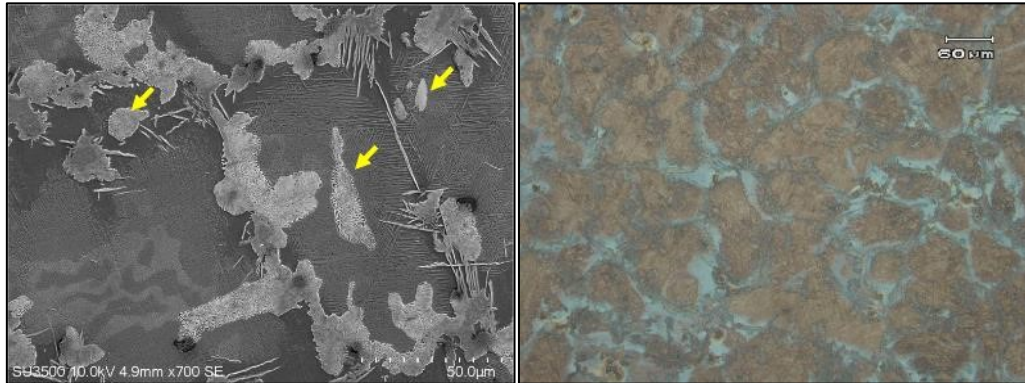


**Figure 7.** Pearlite structure of sample iso HT 600, heated isothermally at 600 °C for 10 H and followed by water quench.

### 3.2. Mechanical properties

Based on the reference, the hardness of manganese steel is about 200 Brinell or convert to Rockwell as 92 HRB [4]. Figure 8 shows the hardness test data for each sample. There is no wide variation in the value of hardness found for the samples, ranging from 90-92 HRB. This hardness is relatively soft because the primary microstructure is Austenite. As-cast samples have higher hardness value due to the presence of carbide. The 600-800 step HT sample also has a relatively higher value than the others because its grain is finer.
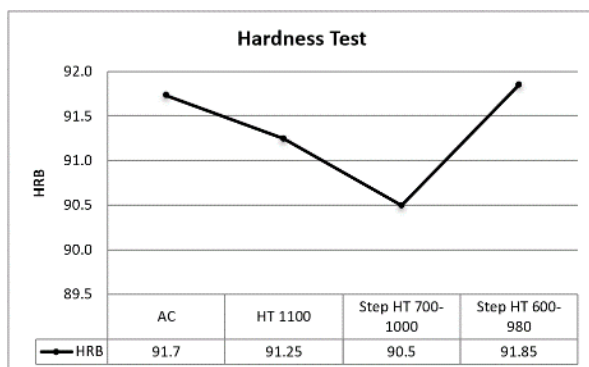


**Figure 8.** Hardness test (rockwell b) result.
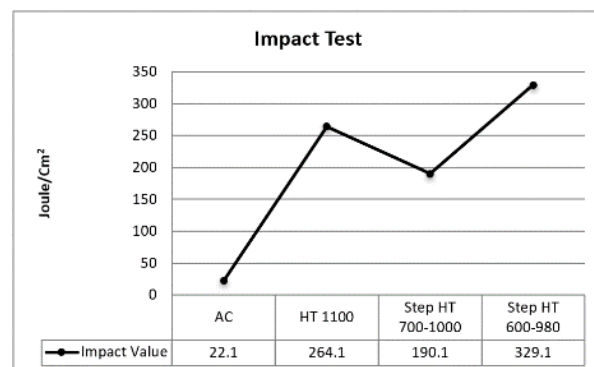


**Figure 9.** Impact test result.

Figure 9 shows the results of the impact testing. It can be discovered that the impact value of the samples increases after the solution heat treatment process compared to the value of the as-cast condition. The low impact value of the as-cast sample is associated with the presence of carbide at the grain boundary, which causes the brittleness of the material. The highest impact value of 329.1 J/Cm$^2$ is observed in Step HT 600-980 sample, and this can be associated with the 100% austenite microstructure supported by the fine grain size of the material. This combination produces good material toughness.

The increasing of the material toughness is related to the smaller grain size, which is produced by step heat treatment. Toughness is the ability of a material to absorb energy without cracking or broken. In most situations, the hardest part of cracking material is nucleating a suitable crack in the first place. When the initial crack is formed, the external forces will propagate the crack. In a grained material, a

crack can only grow as far as the first grain boundary it encounters. After that, it has to either re-nucleate in the adjacent grain or travel along the grain boundary. Traveling along the grain boundary is often an easier path, regarding the energy needed per unit crack area. For small grain, the propagation of the crack requires to deviate around and between many grains, increasing its total area, then the more energy must be dissipated, which makes the material tougher.



|      (a)  As-cast     |     (b)  HT 1100     |   (c) Step HT 700-1000   |   (d)Step HT 600-980   |
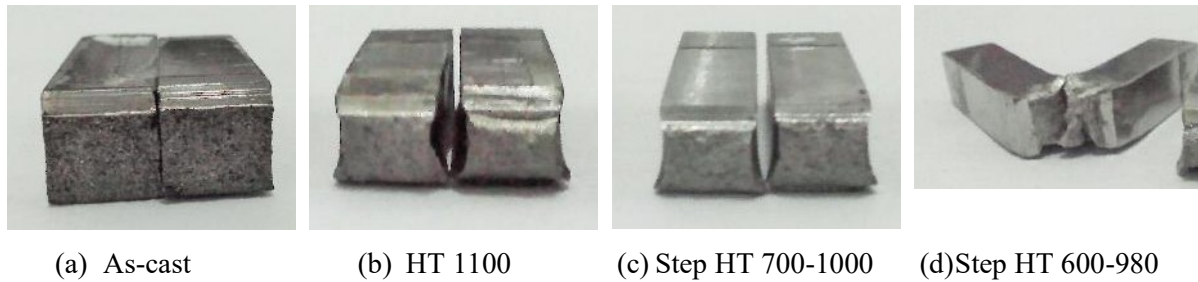
**Figure 10.** Fracture of impact's specimens.

The brittle and ductile behaviour of the material can also be analysed from the type of samples fracture, as shown in Figure 10. The appearance of the impact sample broken piece gives information about the fracture type. The lateral expansion is a measure of the sample ductility. When a ductile metal is broken the test piece deforms before breaking, a pair of 'ears' being squeezed out on the side of the compression face of the sample, as illustrated in Figure 11 the amount by which the sample deforms is measured and expressed as millimetres of lateral expansion [17].
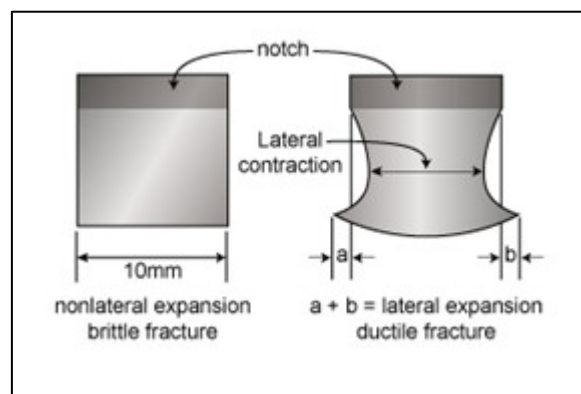


**Figure 11.** Lateral expansion [17].

Figure 10(a) showing the as-cast sample. It indicates the non-lateral expansion fracture, which is shown the brittle fracture. The other samples, which are shown in Figure 10(b) and 10(c), exhibit the pair of 'ears' at its broken piece or lateral expansion condition showing the ductile fracture. The most ductile material is demonstrated by the sample of step HT 600-980 because the sample is not completely broken, as shown in Figure 10(d).

The ductility behaviour of the manganese steel sample is also related to the FCC crystalline structure of manganese steel. FCC structure has a higher packing efficiency of around 74%, and the slip planes are more closely packed than BCC. This condition makes FCC structure easily undergo a large elongation that causes the material tougher.

On the other hand, Step HT 700-1000 sample is observed to have a low impact value compared to the other two heat-treated samples. The low impact value can be as a result of the fact that the pre-heating holding time at 700°C was given for 3 hours, and the holding time at austenitizing temperature

was only 1.5 hours. Because of this, the carbide is not entirely dissolved in the austenite matrix, leaving micro carbide at the grain boundary and causing a decrease in value impact [14, 18].

## 4. Conclusions

From the results of the experiment, it can be concluded that the process of Step HT 600-980 gives the highest impact value of 329.1 J/Cm$^2$. The top result shows that the pre-isothermal heating process at 600°C has a positive effect on producing the new finer grain of pearlite. The fine grain pearlite can still be maintained until the heating to austenitization temperature. The other advantage of the step heating process is that the heating process in the austenitization stage is more efficient than the continuous methods. It was also discovered that the holding time is useful in homogenizing all parts of the sample and produce a final austenite microstructure that is close to 100%. The austenitizing temperature of 980°C with a short holding time of 2 hours proved effective in producing finer grain associated with the maintained fine pearlite grain size, which is formed at the pre-isothermal heating before. Overall the stepped heat treatment process could provide higher toughness, 25 % better, compared to the conventional method of direct heating at the temperatures of 1010°C to 1120°C.

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