Chapter 4

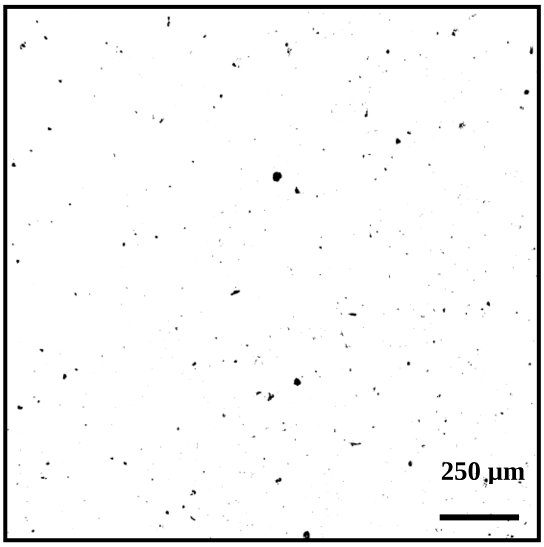
Results and Discussion

4.1. Investigation of alloy 1

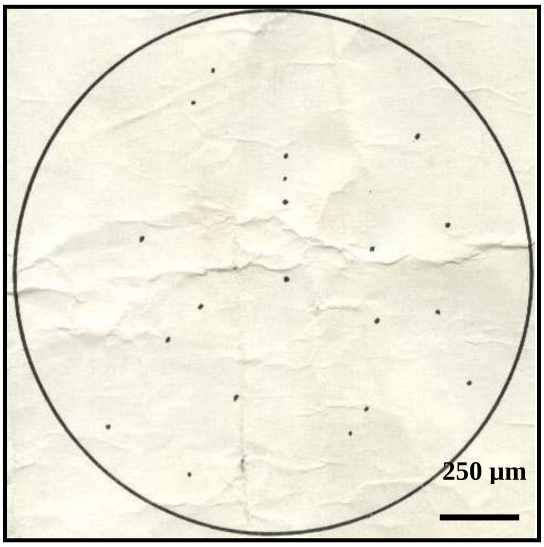
Non metallic inclusions content, microstructure investigation, and mechanical properties like strength, toughness and hardness were investigated.

4.1.1. Non-metallic Inclusion Assessment for alloy 1

The amount, distribution, size and chemical composition of non-metallic inclusions have a direct influence on the steel properties [28].
Non metallic inclusion micrograph of alloy 1 is presented in figure 4.1



a) Actual non metallic inclusions for alloy 1



b) Oxides type of non metallic inclusion according to ASTM

Figure 4.1 Actual non metallic inclusion comparisons for alloy 1 with standard Oxides.

Comparing the micrograph in figure 4.1 (a) with the standard shown in figure 4.1 (b) (according ASTM), it is clear that alloy 1 contains oxides type grade D-2.5-Heavy

It is clear that the non metallic inclusions for alloy 1 that indicated in figure 4.1 are fine, distributed, and homogeneous which would not deteriorates the mechanical properties.

The negative effect of non metallic inclusions can be avoided by controlling its size, and distribution to eliminate the negative effect on the mechanical properties [28].

4.1.2. Microstructure Investigation for alloy 1.

4.1.2.1. As Cast Microstructure

The microstructure shown in figure 4.2 contains martensite matrix with some ferrite aggregates. This microstructure is a heterogeneous microstructure which would lead to low mechanical properties, consequently a suitable heat treatment cycle like quenching and tempering is required in order to make a homogeneous microstructure with better mechanical properties.

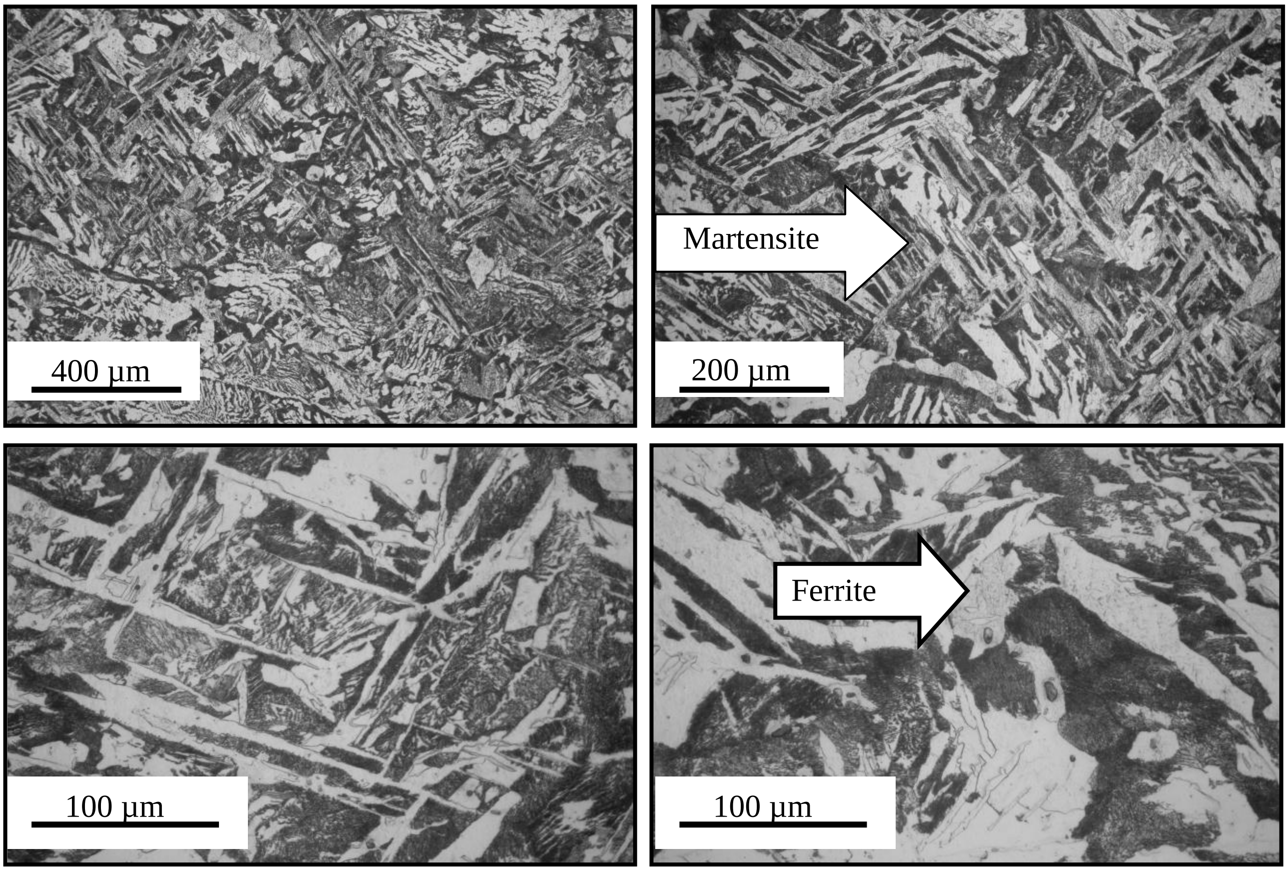


Figure 4.2 The as cast microstructure for alloy 1 with different magnifications

4.1.2.2. Heat treated Microstructure

Figure 4.3 represents the microstructure of alloy 1 after being subjected to hardening and tempering heat treatment cycle.

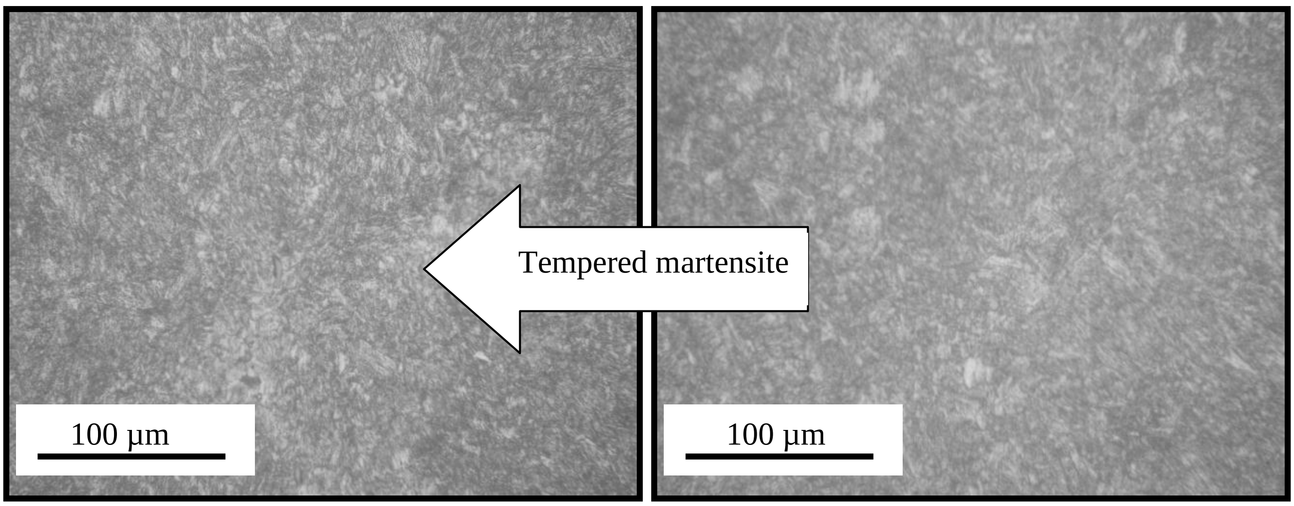


Figure 4.3 Microstructure for Alloy 1 after heat treatment

Quenching process distributed the ferrite portion through the micro structure to form hard martinsite phase which is relatively brittle, so the tempering process is done in order to produce less brittle martinsite (Tempered martinsite) and to relief the internal stresses that occurred during quenching. The microstructure became homogeneous and fine after heat treatment cycle which would lead to high mechanical properties [33].

4.1.3. Mechanical properties evaluation for alloy 1

Engineers are most interested in the way in which metals will respond to the application of external forces. Engineers are dealing with the elastic and plastic behavior, as will as the over-all strength and fracture characteristics of materials. Mechanical properties such as hardness, strength, and toughness are measured for this alloy as follows.

4.1.3.1. Hardness test results

Hardness usually implies a resistance to deformation or resistance to indentation.
The hardness test results are shown in Table 4.1.

Table 4.1 Hardness test results for alloy 1.

Alloy	condition	Specimen number									Mean [HRA]
		1	2	3	4	5	6	7	8	9	
		Rockwell hardness number [HRA]									
Alloy 1	As Cast	51.9	51.1	50.8	49.8	48	47.7	47	45.8	45.7	48.6
	Heat Treated	58.1	58.5	57.7	58.8	57.3	57.6	58.9	56.5	58.7	58

The hardness of the all specimens under testing was increased after the heat treatment operation. The hardness of alloy 1 is increased by 9.4 [HRA]. So the heat treatment cycle (hardening – tempering) has better significant effect on hardness.
Figure 4.4 confirms the good effect of heat treatment on hardness.

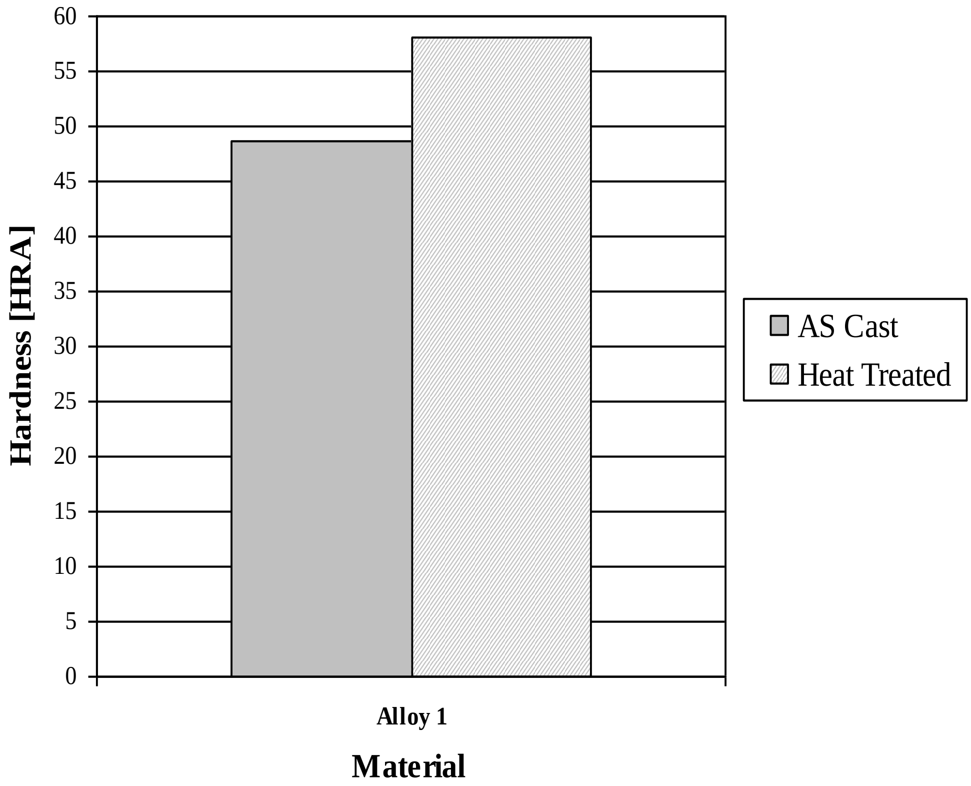


Figure 4.4 Hardness results for alloy 1

4.1.3.2. Tensile test results

Specimens were tensile tested from each alloy before and after the heat treatment. The ultimate tensile strength (UTS) can be clearly calculated whereas the yield is considered as 0.80% of the ultimate tensile strength.

Table 4.2 presents the actual tensile test properties of alloy 1 for the as cast and heat treated conditions.

Table 4.2 Tensile properties for alloy 1

Alloy	Condition	Specimen number	Tensile mechanical properties	
			UTS [MPa]	Mean UTS [MPa]
Alloy 1	As cast	1	468.79	466.9
		2	464.97	
		3	466.83	
	Heat treated	1	616.56	603.9
		2	592.70	
		3	602.43	

the tensile properties presented in figure 4.5 for the as cast and heat treated conditions confirm what have been obtained from the hardness measurements ensuring that the heat treatment cycle was proper selected, the present results are in agreement with the results obtained from the ASTM A352 standard. The improvement of the mechanical properties which is accompanied by the heat treatment cycle is attributed to the homogenous and fine microstructure.

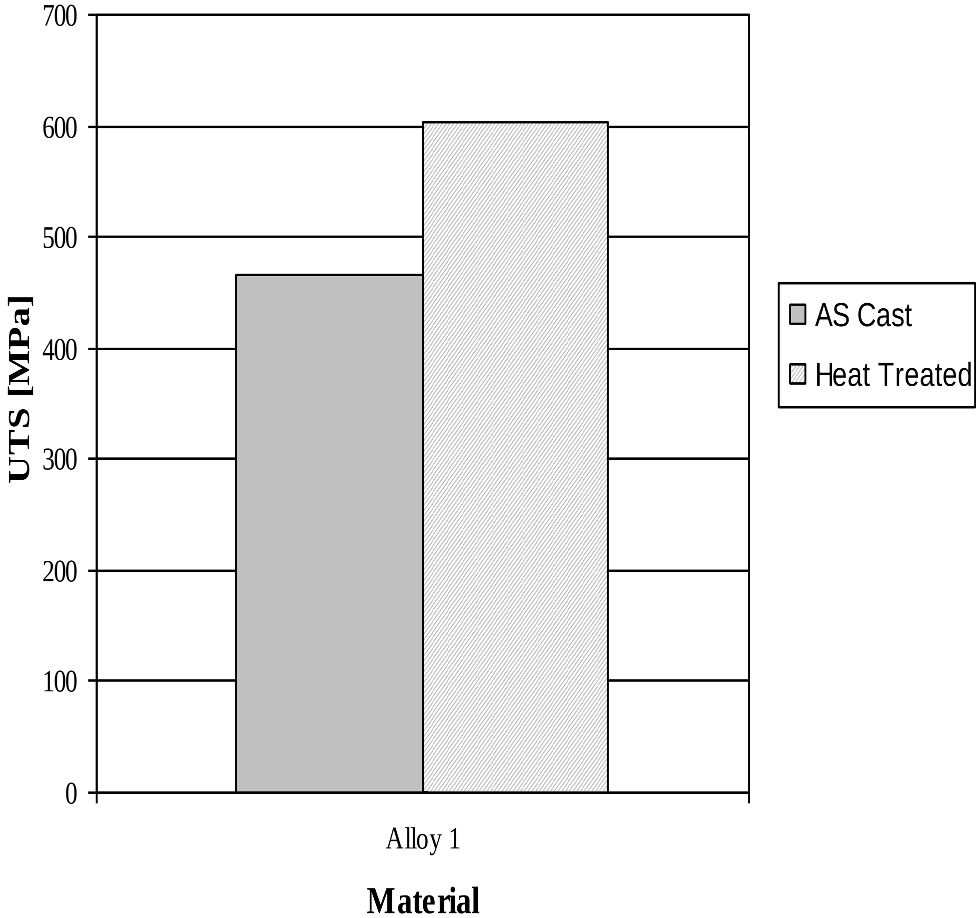


Figure 4.5 Tensile test results for alloy

4.1.3.3. Impact test results

The energy values presented at the following tables are the mean of three test specimens at least to insure that the values are matched with each others.
All specimens are marked with suitable code to be able to identify each one after testing to make the fractographic investigation.
Impact values before and after heat treatment at different subzero temperatures for alloy 1 are presented in table 4.3.

Table 4.3 Impact properties for Alloy 1

Condition	Test temperature [^oC]	Mean energy value [J]
As Cast	Room temp. (27 ^oC)	17.2
	-20	8.9
	-40	7.3
	-60	5
	-73	4.4
Heat Treated	Room temp. (27 ^oC)	61.5
	-20	54.8
	-40	42.8
	-60	38.4
	-73	25.2

The data included in table 4.3 is presented in figure 4.6 for as cast and heat treated conditions.

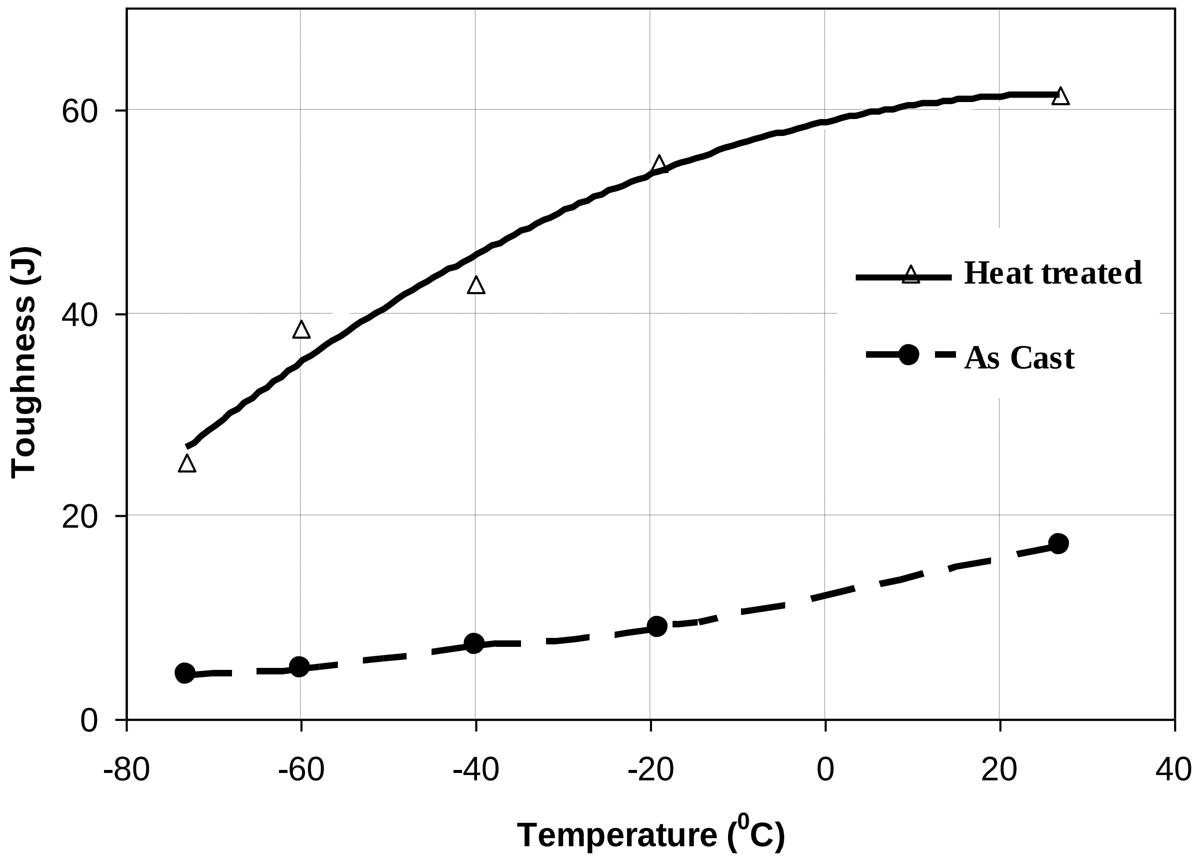


Figure 4.6. Impact toughness for alloy 1 before and after heat treatment.

It is clear that the impact value at room temperature for the as cast conditions has lower value (17.2 J) than that that of heat treated condition (61.5 J). This is clearly reflecting the successful effect of heat treatment cycle and confirms what have been concluded previously [33].

On the other hand, it is observed that toughness decreased continuously with the decreasing of testing temperature for both conditions.

Further more the toughness of the heat treated condition is always higher than the as cast condition which is another positive effect of the heat treatment cycle.

Generally, when the ambient temperature drops, the toughness of materials also decrease and it becomes very low at a certain low temperature. This is called cold brittleness, and the temperature at which the material turns from a tough state to a brittle state is called the ductile–brittle transition temperature [4].

Steel castings that have been quenched and tempered have higher notch toughness than similar castings in the as cast conditions [10].

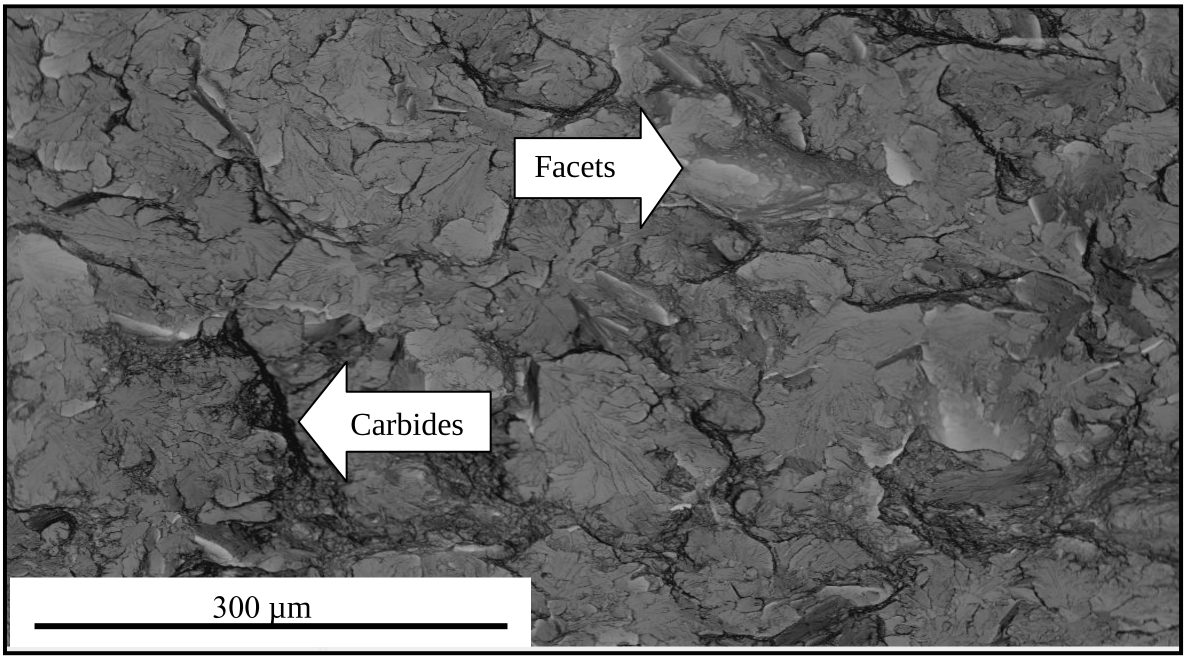
4.1.4. SEM Fractographic Investigation for alloy 1

Fracture surface of impact specimens give the right guide to the investigation weather the material followed ductile behavior or brittle behavior during the testing conditions.

4.1.4.1. As Cast SEM Fractographic Investigation

Fracture surfaces of the dynamically deformed and failed samples were examined in a scanning electron microscope (SEM) so as to determine the macroscopic fracture mode, and to concurrently characterize temperature influences on fine-scale topography. Figure 4.7 (a-b) illustrates the fracture surface of impact specimen at different magnifications for the as cast stat tested at room temperature.

a)



b)

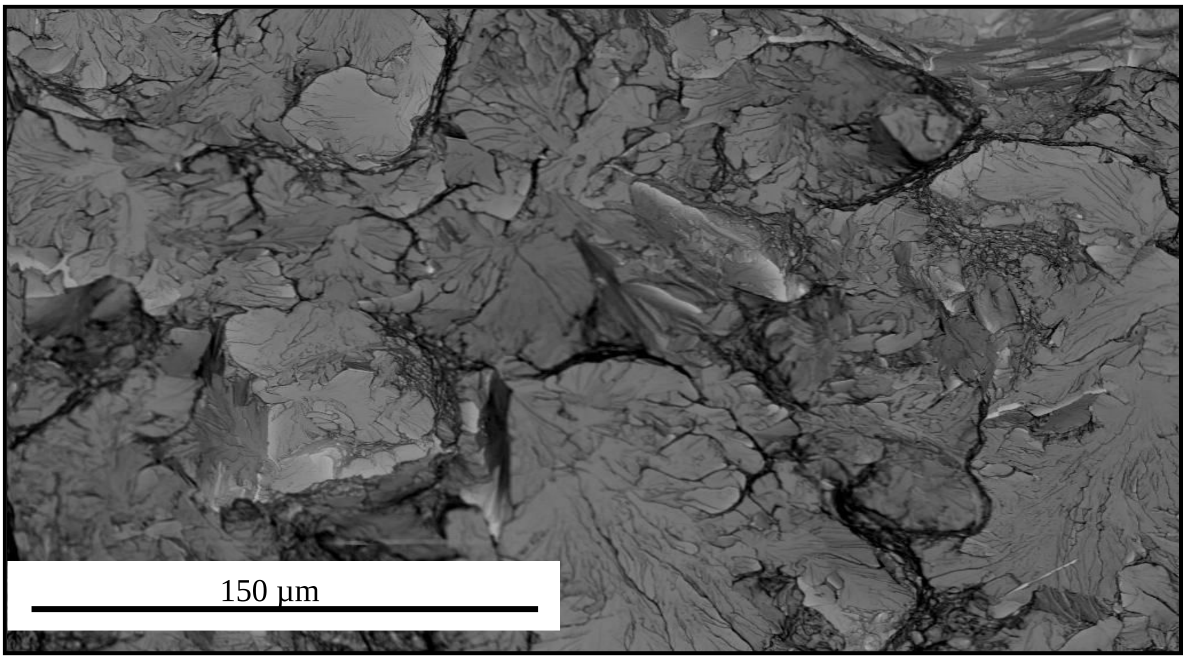


Figure 4.7 (a-b) SEM Fractographic for Alloy 1 at Room Temperature for as cast state.

A vast majority of the fracture surface is cleavage facets with thin layers of carbides imbedded between the facets. This fracture pattern reflects the low value of toughness at the room temperature; the fracture patterns confirm the impact as well as the other mechanical testing results.

For more detecting of the embedded carbide phase, high magnification [X 1500] was taken on the fracture surface as indicated by figure 4.8. It is noticeable that the carbide layer has a continuous feature determining the impact properties and some dimples appeared with facets.

The continuous carbide film forms net-like shape, which facilitates the crack propagation.

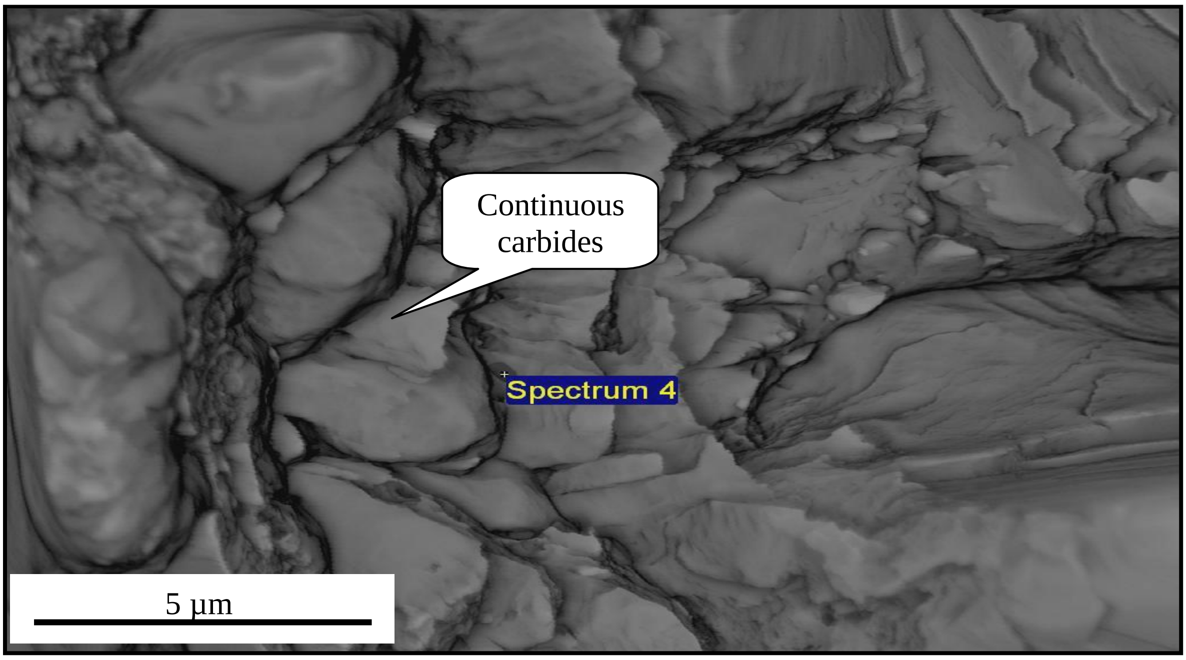


Figure 4.8 SEM for Alloy 1 at Room Temperature in as cast stat with high magnification

For more details an investigation on the fracture surface, XRD qualitative analysis was applied on the imbedded carbide.

Figure 4.9 and table 4.4 represents the XRD chart and qualitative chemical contents of the carbide layer. It can be summarized that the alloy contains iron carbide which is brittle.

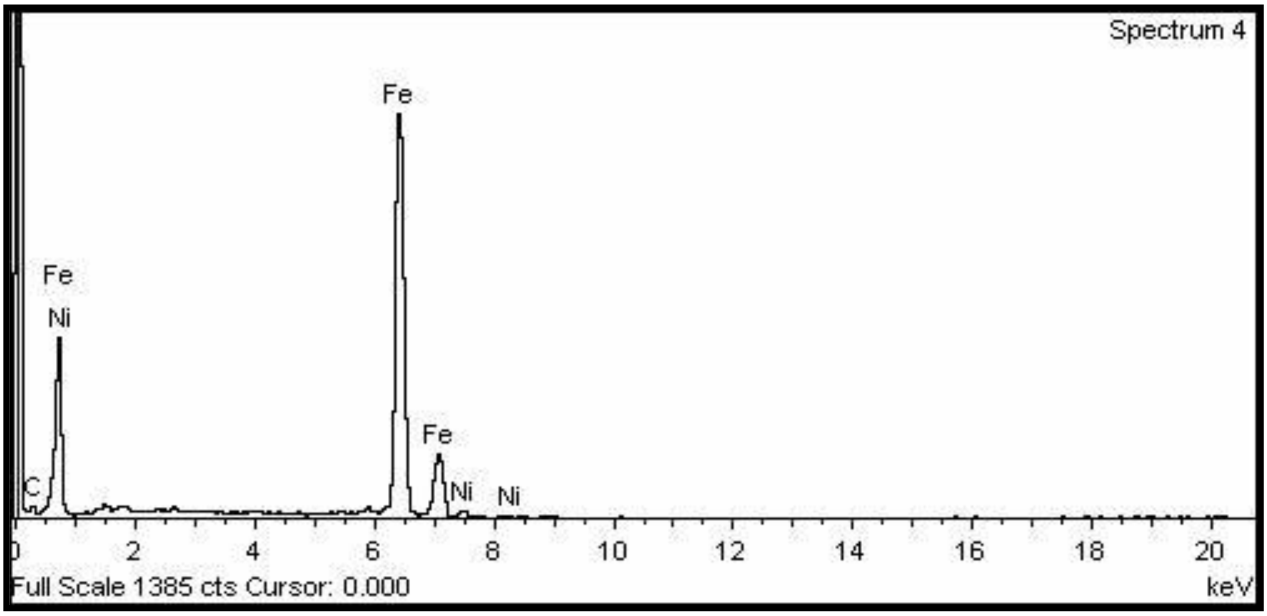


Figure 4.9 XRD for Alloy 1 before heat treatment (as cast)

Table 4.4 XRD Analysis for Alloy 1 before heat treatment (as cast)

Element	C	Fe	Ni
Weight%	6.31	91.26	2.43
Atomic%	23.86	74.26	1.88

Figure 4.10 illustrates the fracture surface for impact specimens tested at -60 ^{0}C.

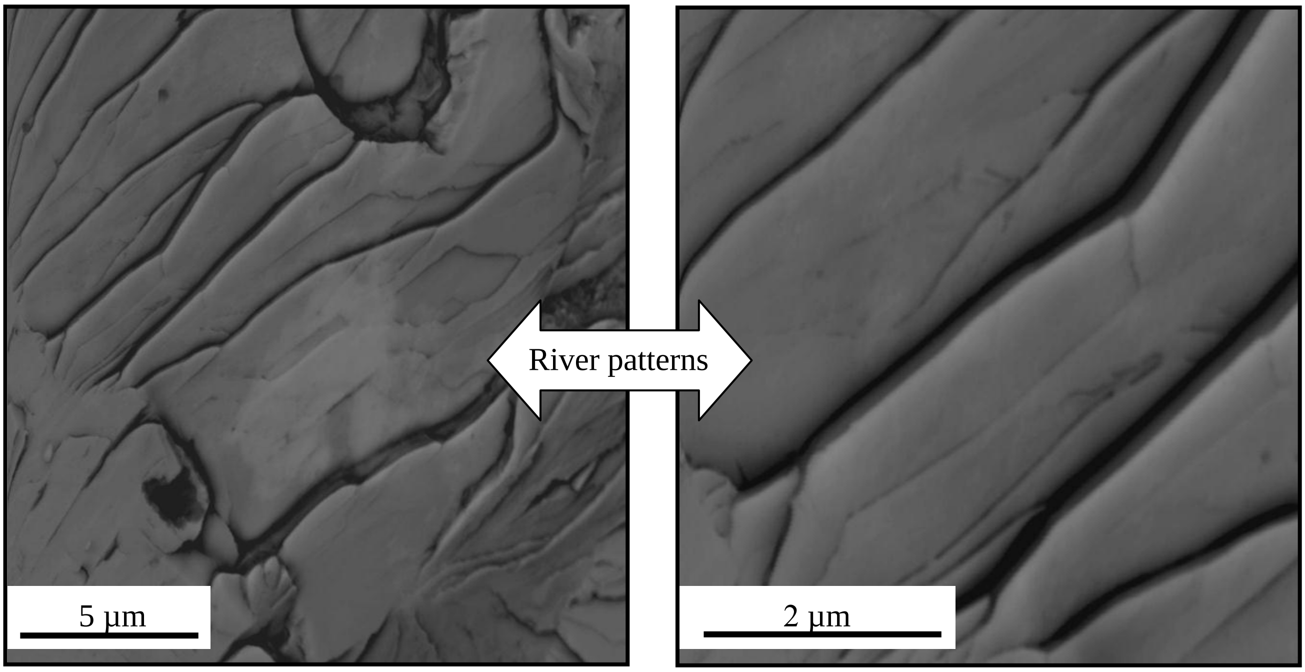


Figure 4.10 SEM Fractographic for Alloy 1 at -60^{o}C at different magnifications for the as cast state.

Both graphs show river pattern fracture. The impact results will be highly decreased as a result of lowering the testing temperature.

4.1.4.2. Heat treated SEM Fractographic Investigation

Heat treatment processes are considered as a powerful tool to improve and develop better mechanical properties.
The fracture surface of a quenching-tempering impact specimen at room temperature is presented in figure 4.11.

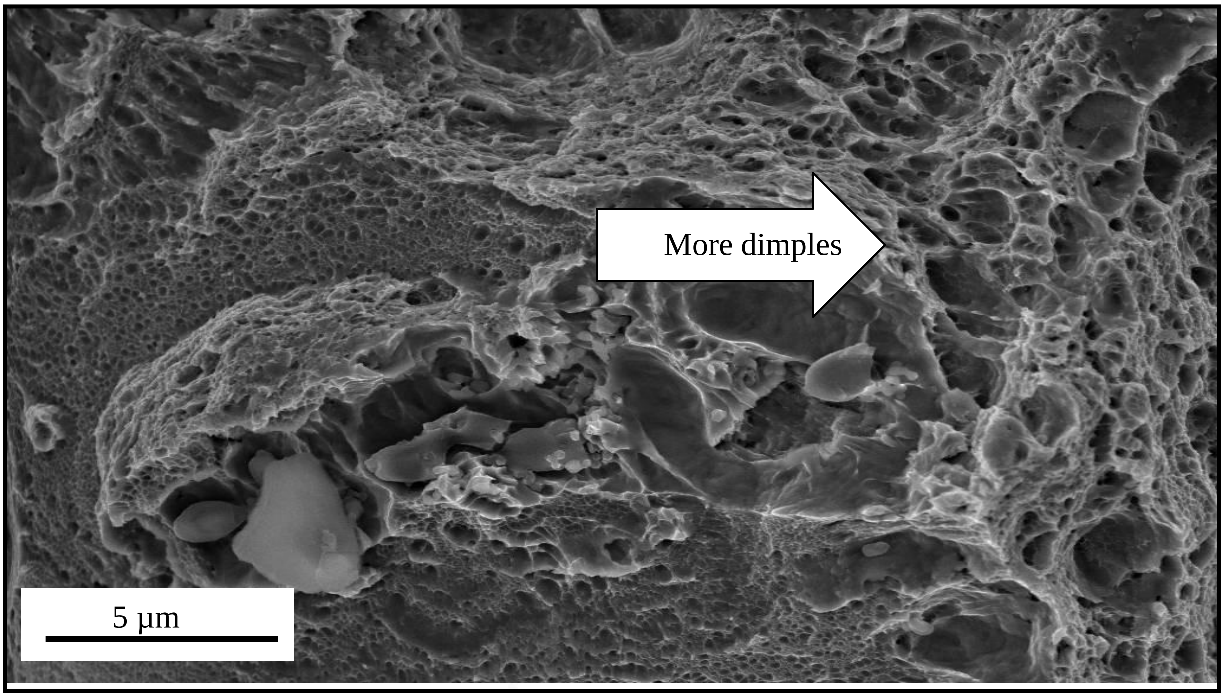


Figure 4.11 SEM Fractographic for Alloy 1 at Room Temperature after heat treatment

The fracture surface becomes dimples rupture farther more, it becomes fine dimples which reflect the increasing of toughness after the applied heat treatment cycles.
However, there are some carbide tracing concentrated at the route of that dimples forming troughs.
It is obvious that the fracture mode is changed from brittle facets with imbedded continuous carbide to ductile fine dimples. The fractograph confirms the positive value added by the heat treatment where these fine dimples show more ductility [33].

It is beneficial to look on the fracture heat treated specimens at lower temperature than room temperature to expect the effect of low temperature on the heat treated alloy 1.
Figure 4.12 represents the fracture mode of heat treated alloy 1 at -40 ^{0}C.

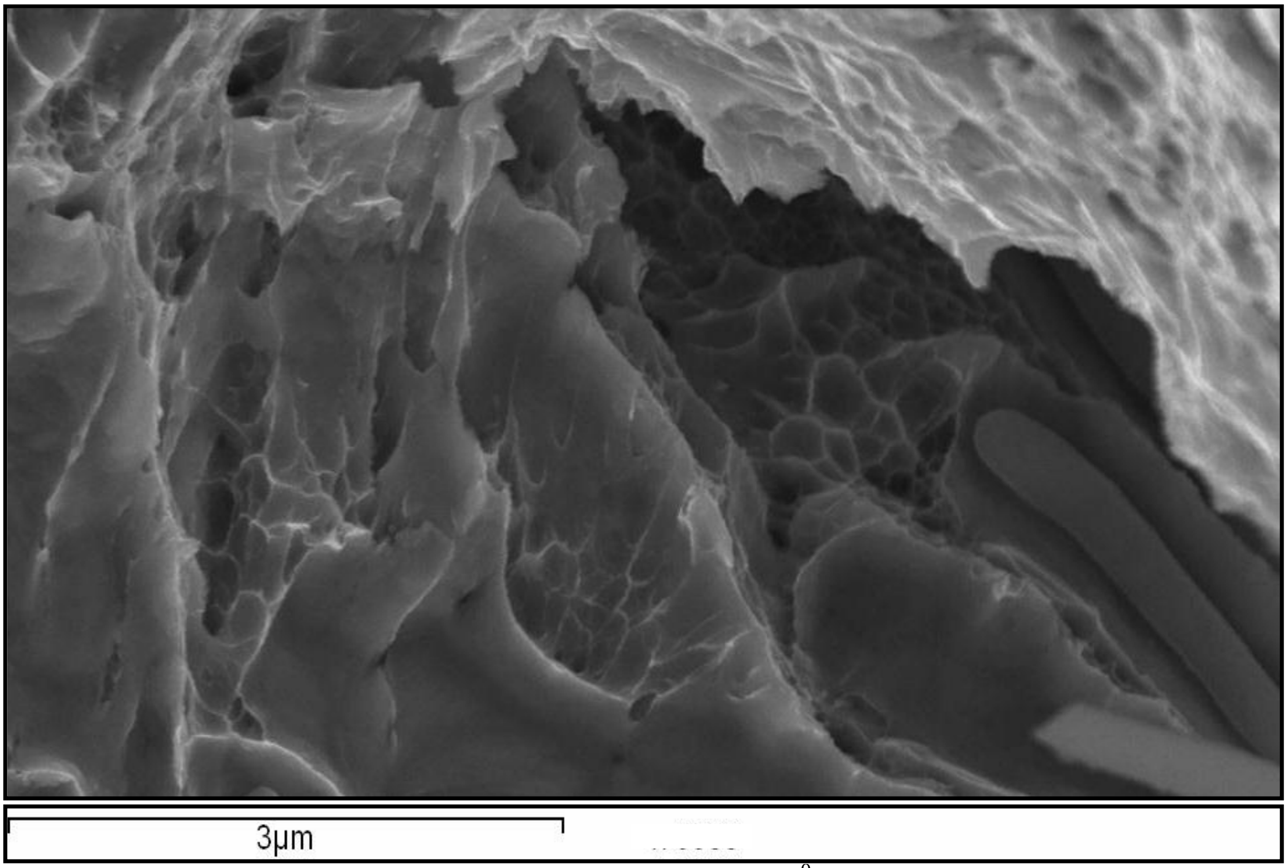


Figure 4.12 SEM fractographic for Alloy 1 at -40 ^{0}C after heat treatment

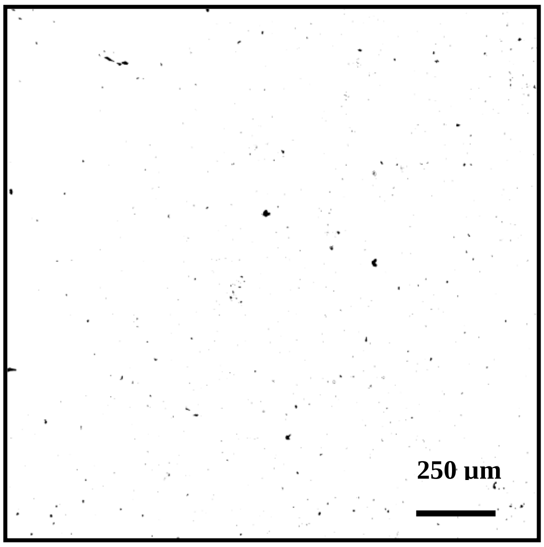
The fracture mode goes towards brittle behavior, as the micrograph changed from complete fine dimples at room temperature to mixture of dimples with considerable amount of facets. However, the present condition does not mean high brittle steel behavior. The dimples portion determines the ductility level.

4.2. Investigation of alloy 2

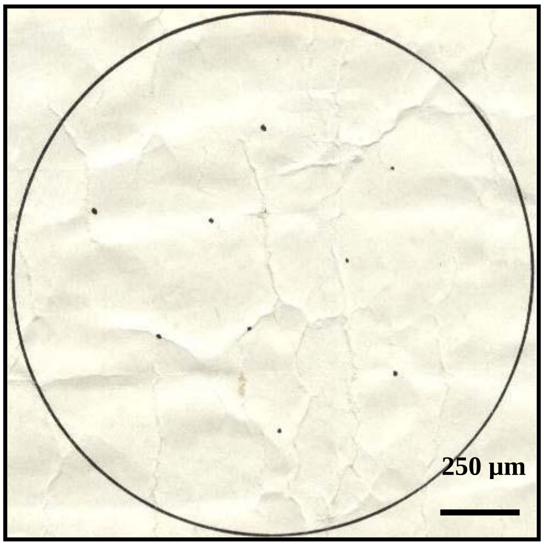
Chromium and molybdenum were added to the rest of the melt in order to obtain higher strength than that achieved by the conventional alloy 1. Recently, a work published on TMS 2010 confirms that addition of both elements increase strength by making benefit of solid solution hardening and precipitation hardening [43].

4.2.1. Non metallic inclusions in alloy 2.

Figure 4.13 represents the actual non metallic inclusion of alloy 2 compared with the standard oxide of ASTM.



a) Actual non metallic inclusions for alloy 2



b) Standard oxides type of non metallic inclusion according to ASTM

Figure 4.13 Actual non metallic inclusion comparisons for alloy 2 with standard Oxides.

The composition reveals that non metallic inclusions in this alloy are Oxides of grade D-1.5-T, which are not highly harmful and accepted. The non metallic inclusions are thin and homogenously distributed. The mechanical properties are slightly affected by these inclusions [28].

4.2.2. Microstructure Investigation for alloy 2.

4.2.2.1. As cast microstructure

After etching the as cast specimen with 2 pct nital (2%Nitric acid plus 98% Alcohol), microstructure of alloy 2 can be clearly shown in figure 4.14.

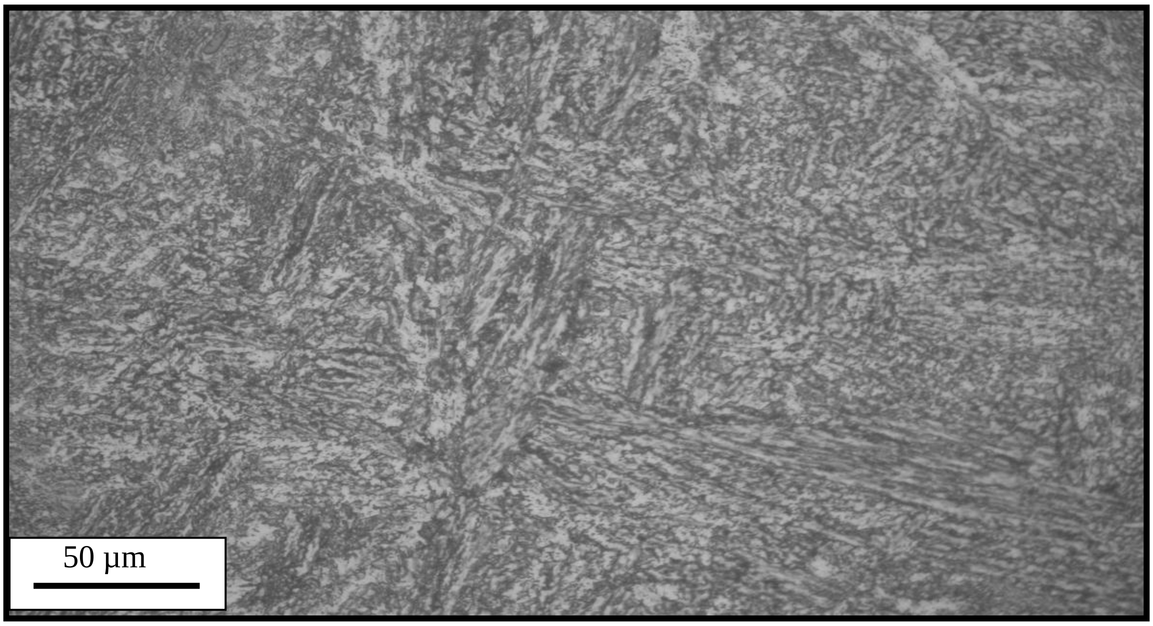


Figure 4.14 the as cast Microstructure for Alloy 2

A very fine martensitic structure shown in figure 4.14 results due to the early forming of Molybdenum carbide (Mo_3C) and Chromium carbide (Cr_3C) during solidification as it was confirmed by reference [43], these carbides are working as nuclei or seeds which would lead to more fine martensitic grains.

4.2.2.2. Heat treated Microstructure

A heat treatment cycle (quenching-tempering) has been applied on the developed alloy 2 for comparison between alloy 1 and alloy 2.

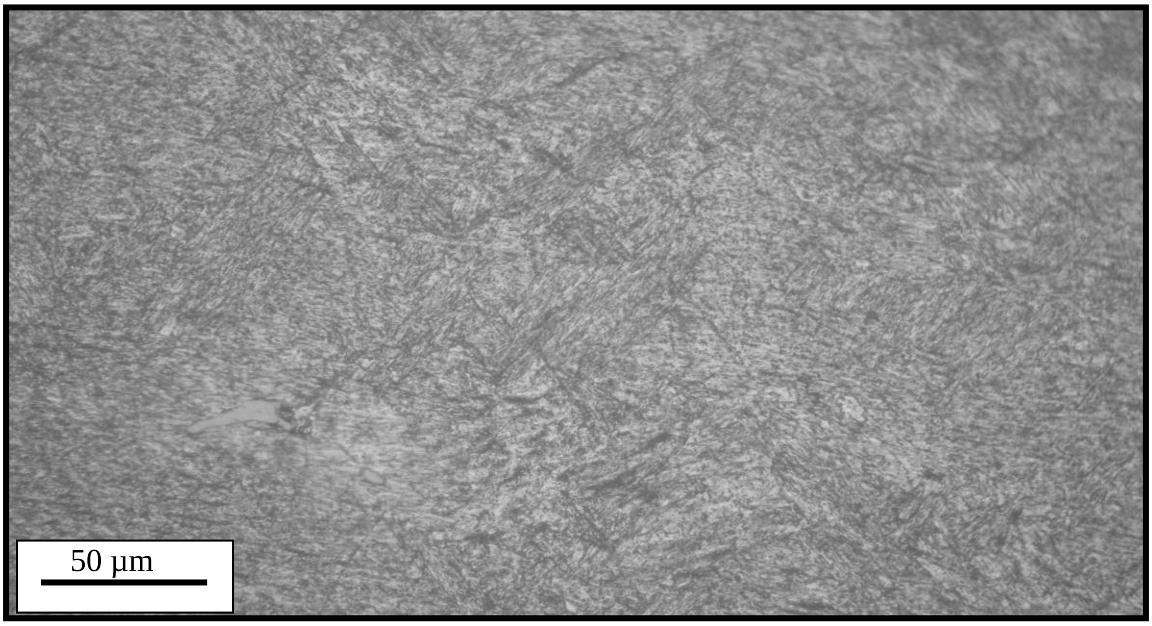


Figure 4.15 Microstructure for Alloy 2 after heat treatment

The heat treated microstructure is slightly finer that in the as cast state, so from figure 4.14 and figure 4.15 there is no significant effect in the microstructure after the heat treatment..

4.2.3. Mechanical properties evaluation for alloy 2
4.2.3.1. Hardness test results

Table 4.5 contains hardness values measured on alloy 2 in the as cast and heat treated conditions. Farther more, the main values of hardness are presented in figure 4.16.

Table 4.5 Hardness test results for alloy 2.

Alloy	condition	Specimen number									Mean [HRA]
		1	2	3	4	5	6	7	8	9	
		Rockwell hardness number [HRA]									
Alloy 2	As Cast	67.3	66.6	66.4	66	65.8	65.4	64.5	63.8	61.7	65.3
	Heat Treated	66.4	65.7	66.8	66.7	67.4	66.4	65.9	65.8	66.1	66.4

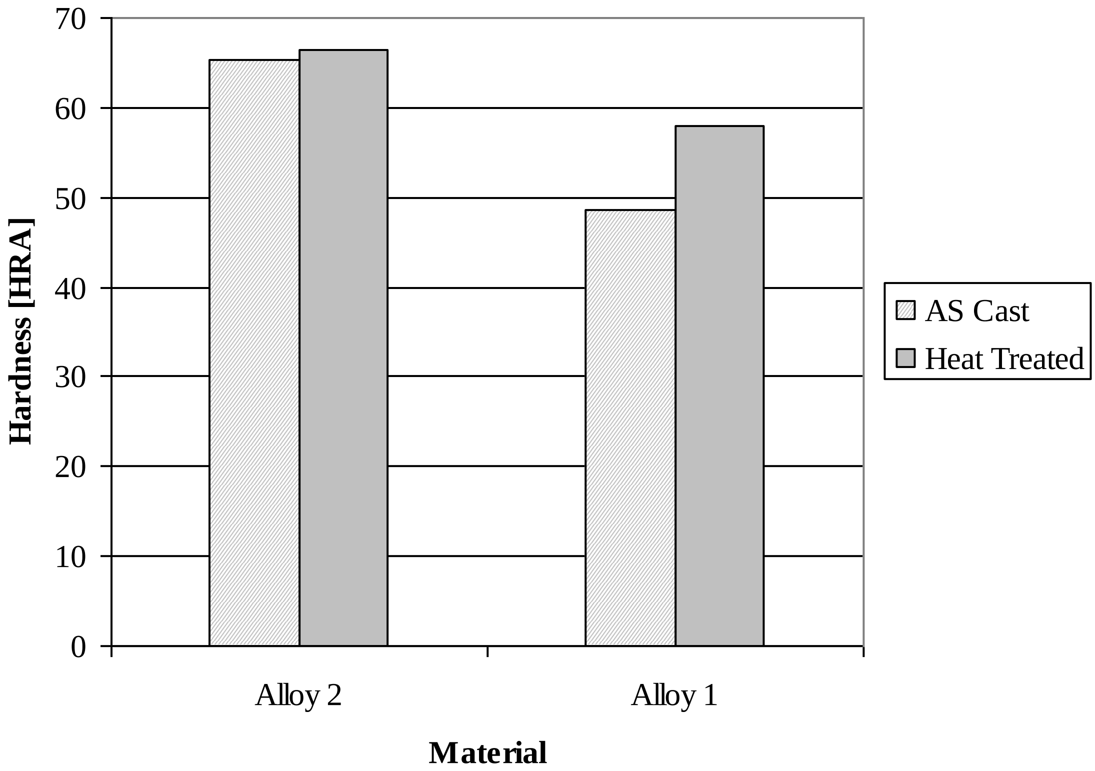


Figure 4.16 Hardness test results for alloy 2 compared with alloy 1

Hardness was increased by a non significant value which indicates that the heat treatment was not effective as in alloy 1.
From the comparison between the hardness results of alloy 1 and the investigated alloy 2, it is clear that the hardness results increased from 48.6 HRA in alloy 1 to 65.3 HRA in the second alloy 2 in the as cast state due to the addition of Chromium and Molybdenum, Which was recently confirmed by the work published in TMS 2010 [43].

4.2.3.2. Tensile test results

The engineering tension test is widely used to provide basic design information on the strength of materials.

Tabulated tensile results are indicated in table 4.6 for both as cast and heat treated states of alloy 2. Mean ultimate strength is illustrated in figure 4.17. It is clear that strength was not increased but it is incremental decrease which confirms that the commercial heat treatment cycle (quenching-tempering) was not recommended. In some other work published [11], it was recommended to carry out tempering at 650 ^{0}C and aging for 64 hours at 425 ^{0}C.

Table 4.6 Tensile properties for alloy 2

Alloy	Condition	Specimen number	Tensile mechanical properties	
			UTS [MPa]	Mean UTS [MPa]
Alloy 2	As cast	1	911.46	903.4
		2	903.18	
		3	895.56	
	Heat treated	1	860.70	846.55
		2	846.55	
		3	832.40	

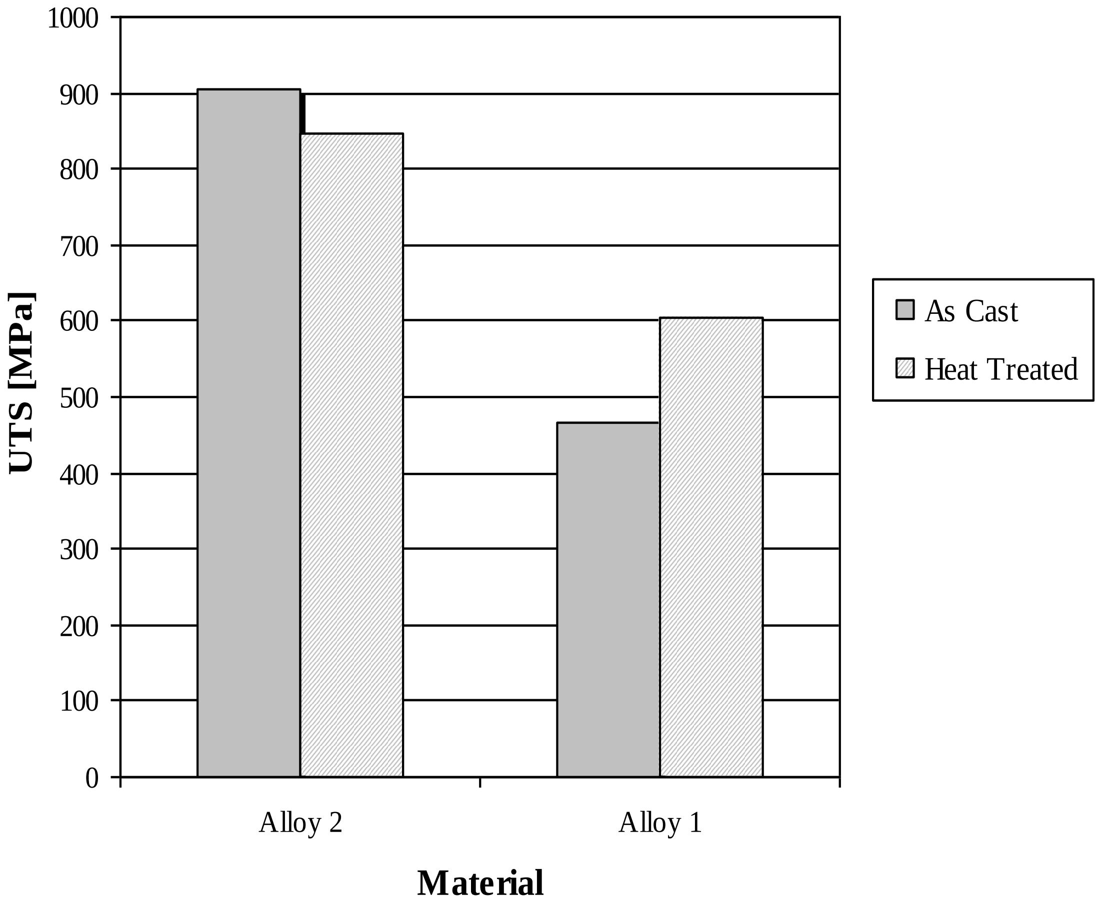


Figure 4.17 Tensile test results for alloy 2 compared with alloy 1

The comparison between the ultimate results of alloy 2 and alloy 1 clearly confirms the positive effect of Chromium and Molybdenum addition in raising the strength of alloy 2.

4.2.3.3. Impact test results

The impact tests have been carried out at different temperatures. The impact results are also tabulated and presented in table 4.7 and figure 4.18.

Table 4.7 Impact properties for alloy 2

Condition	Test temperature [^{o}C]	Mean energy value [J]
As Cast	Room temp. (27 ^{o}C)	13.2
	-20	8.9
	-40	7.35
	-60	6.25
	-73	5.2
Heat Treated	Room temp. (27 ^{o}C)	13
	-20	10.2
	-40	5.3
	-60	4
	-73	3.8

It was noticed that the impact values after heat treatment cycle changed positively at the region of temperature from 27 [^{o}C] to -30 [^{o}C].
The heat treatment has a negative effect on the impact values at the other region (from -30 ^{o}C to -73 ^{o}C) as shown in figure 4.18.

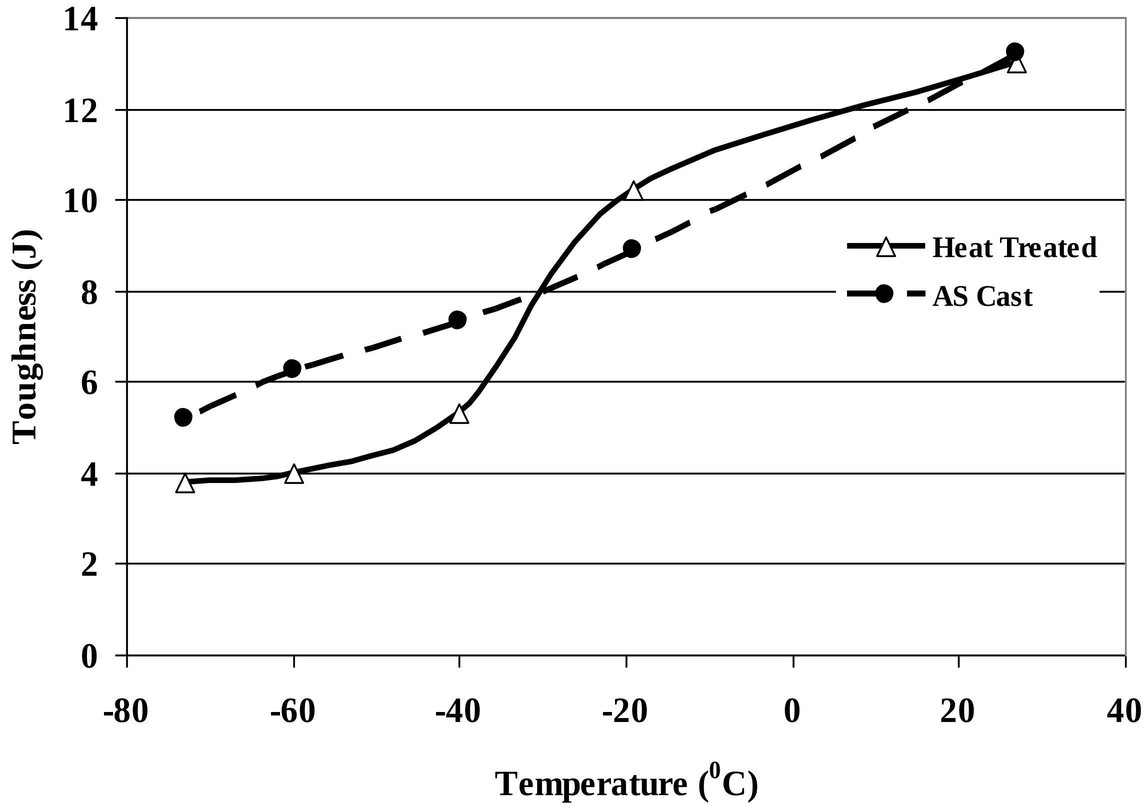


Figure 4.18 Impact toughness results for alloy 2 before and after heat treatment

Obviously, the inadequate heat treatment accelerates the ductile- brittle transition temperature (ITT) at about -30 ^{o}C.

The impact results comparison between the investigated alloy 2 and alloy 1 is presented in figure 4.19.

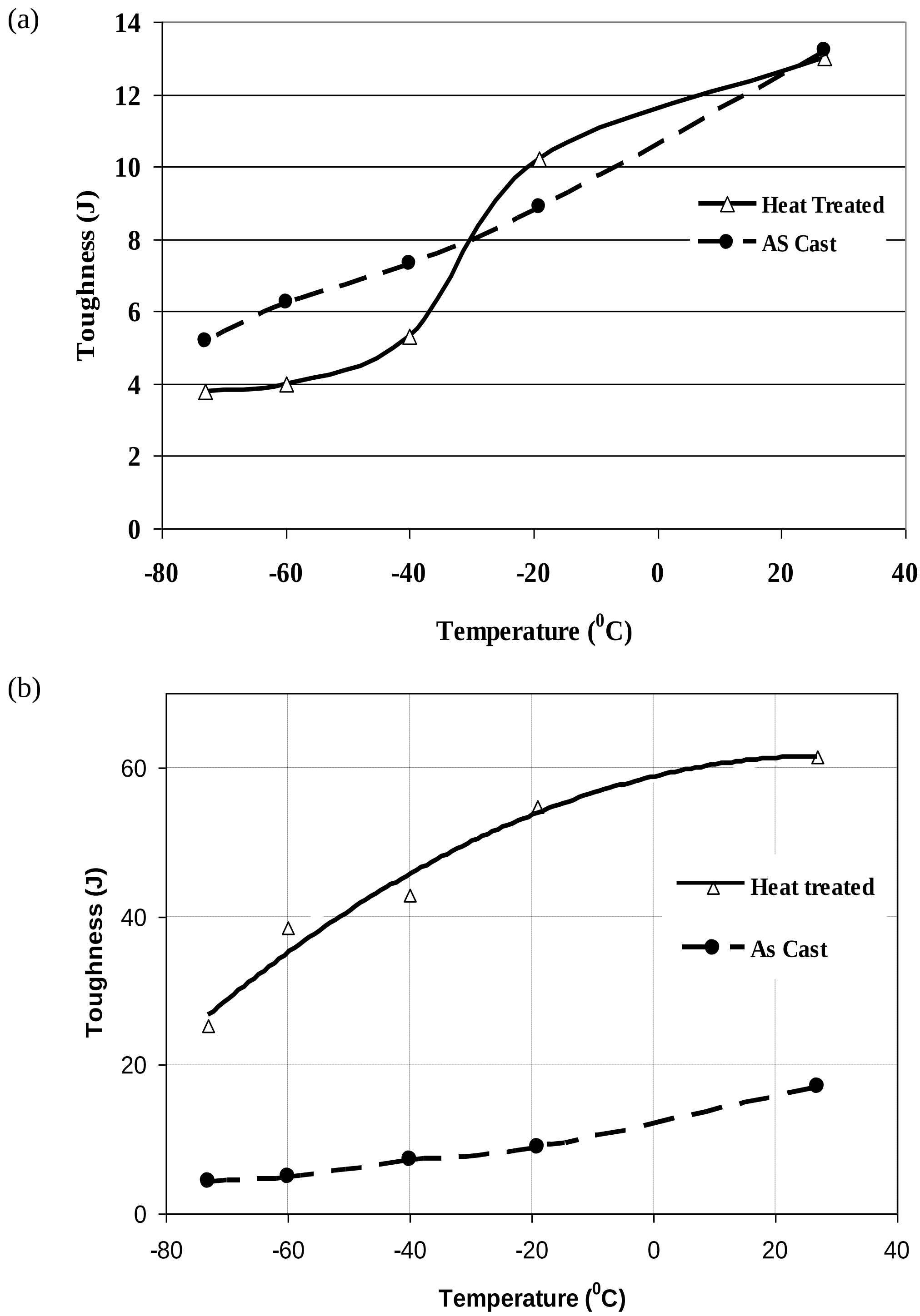


Figure 4.19 (a) Actual impact results of alloy 2 (b) Actual impact results of alloy 1

The impact results of the as cast stat are nearly identical for both alloys. Contrary the impact values of alloy 1 are higher than that of alloy 2 after the heat treatment cycle. The previous results emphasis that the quenching-tempering heat treatment cycle is necessary for alloy 1 and not recommended for alloy 2 [11].

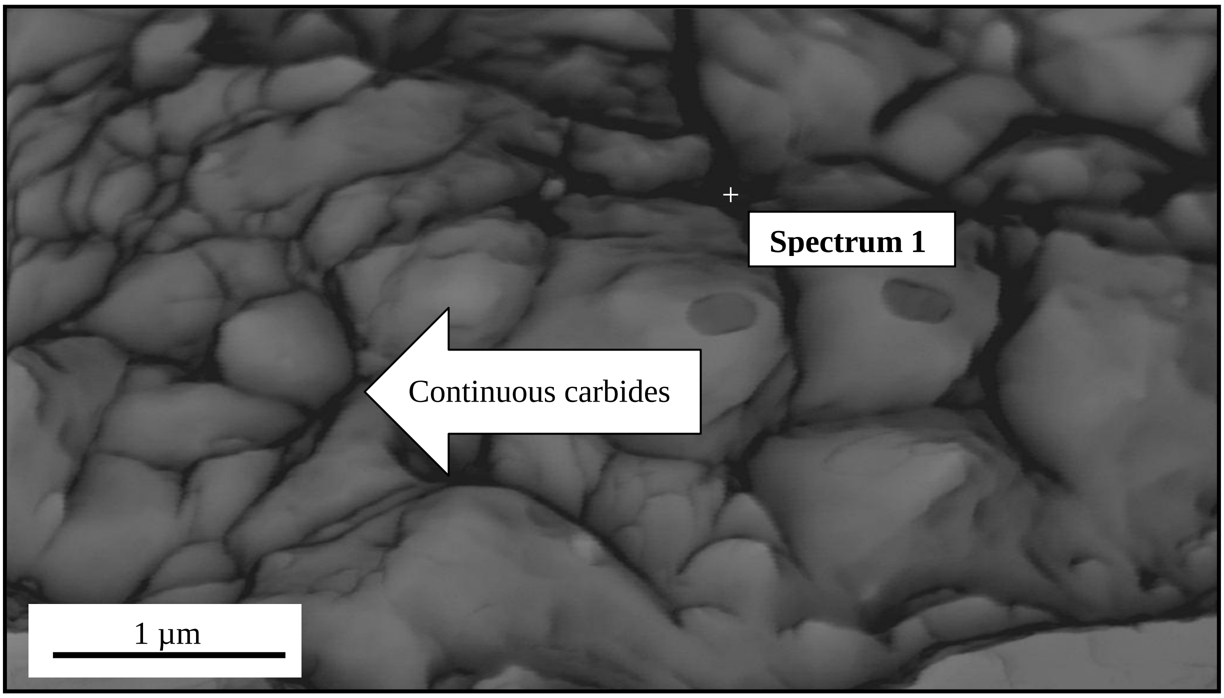
4.2.4. SEM Fractographic Investigation for alloy 2

Fracture surface are considered as an accreditation of the measured mechanical properties.

4.2.4.1. As Cast SEM Fractographic Investigation

Figure 4.20 represents the fracture of impact test specimen tested at room temperature. The fracture surface shows coarse dimples rupture and continuous carbide film impeded between this dimples. Dimples reflect the impact value at room temperature and the continuous impeded carbide film reflects also the low impact value at that temperature.

A qualitative x-ray analysis (XRD) has been performed on the carbide film. Figure 4.20 contains the XRD pattern and table 4.8 includes the peak values of the containing elements. The XRD pattern states that Chromium carbide was found.



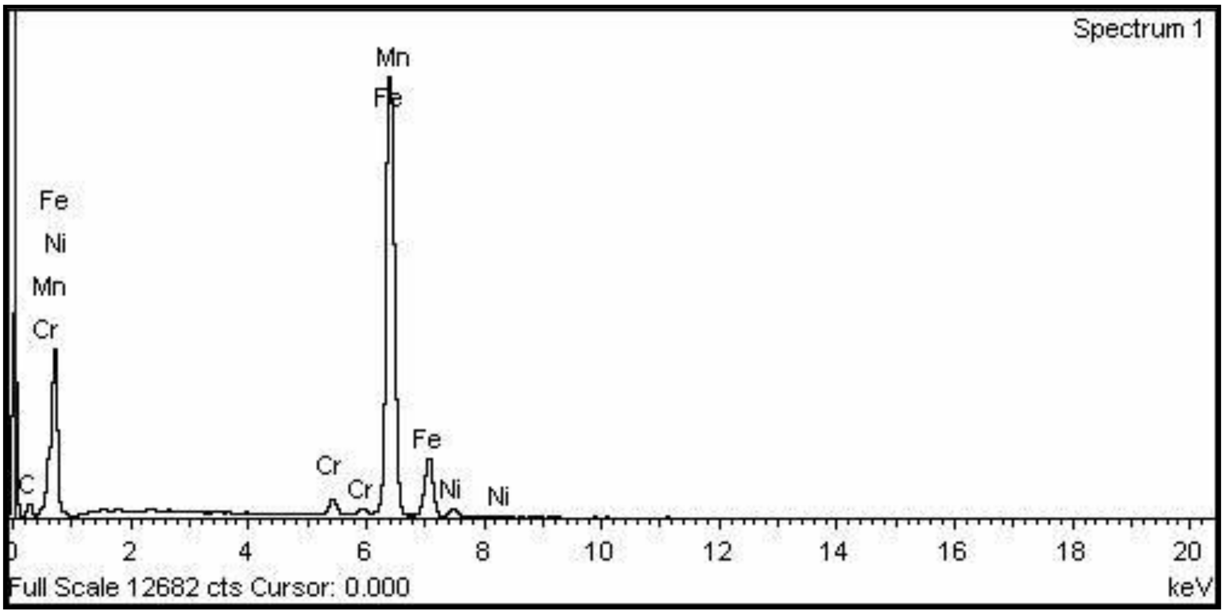


Figure 4.20 SEM Fractographic for Alloy 2 at Room Temperature before heat treatment (as cast)

Table 4.8 XRD Analysis for Alloy 2 before heat treatment

Element	C	Cr	Mn	Fe	Ni
Weight%	6.84	1.69	0.67	88.37	2.43
Atomic%	25.44	1.45	0.54	70.71	1.85

Figure 4.21 represent the fracture surface of a specimen of alloy 2 which was tested at -50 ^{0}C.

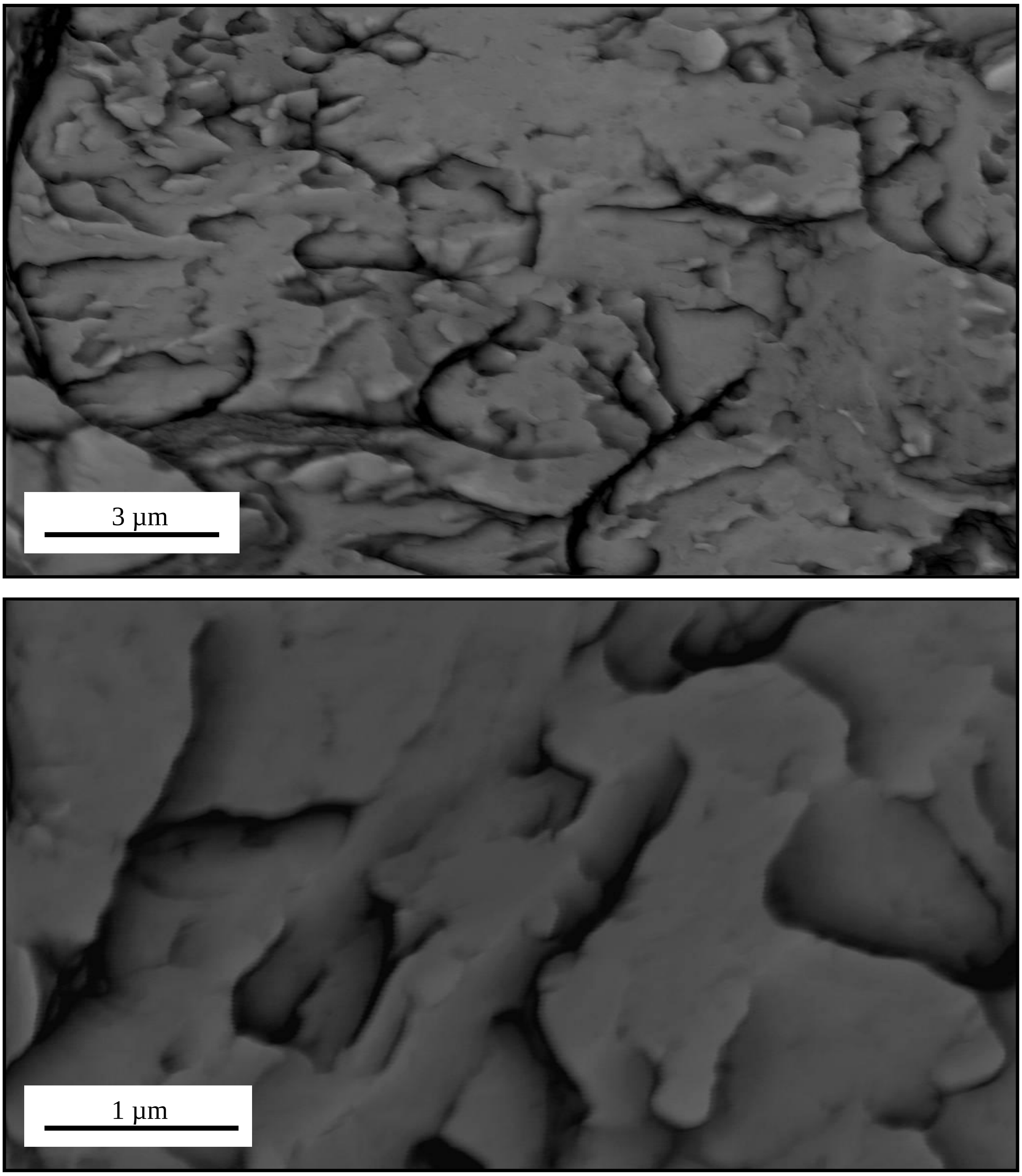


Figure 4.21 SEM Fractographic for Alloy 2 tested at -50 ^{0}C before heat treatment (as cast)

Low test temperature changed the ductile dimples mode at room temperature into complete facets indicating cleavage fracture.

4.2.4.2. SEM Fractographic Investigation after heat treatment for alloy 2

Figure 4.22 illustrates the fracture surface after the heat treatment cycle (quenching-tempering)

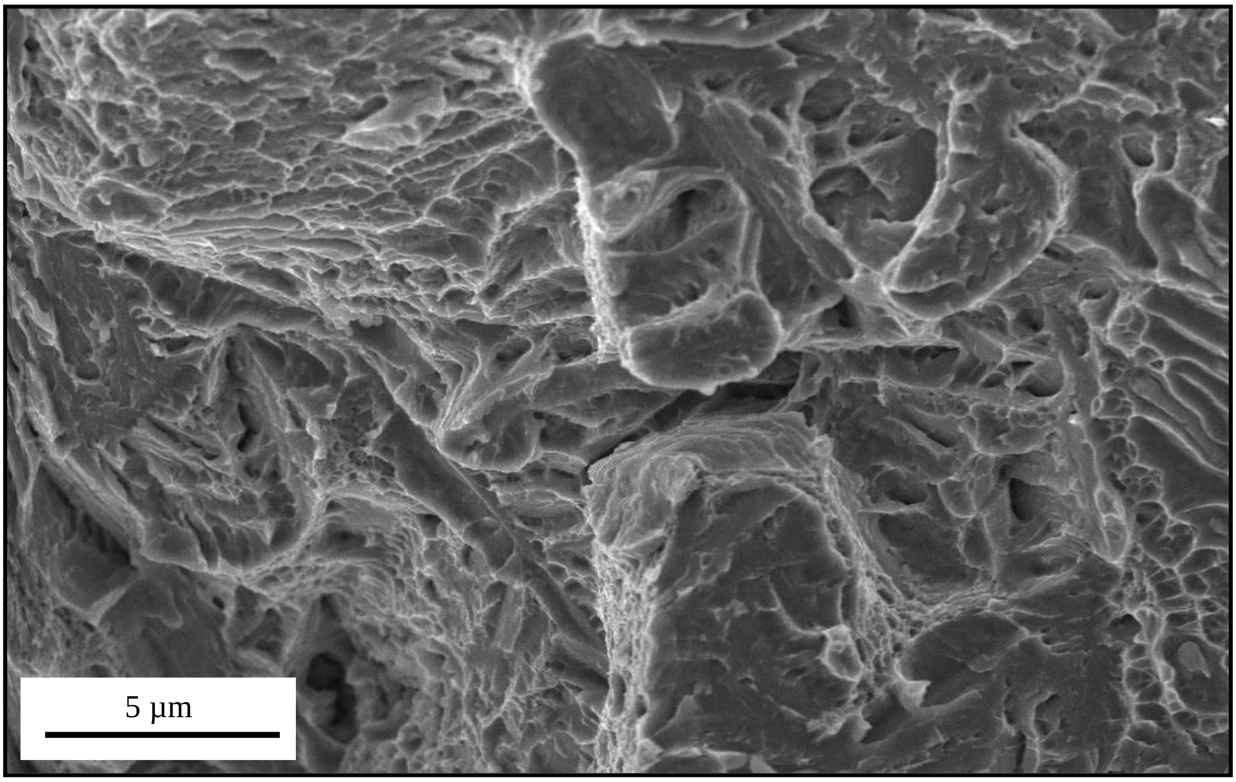
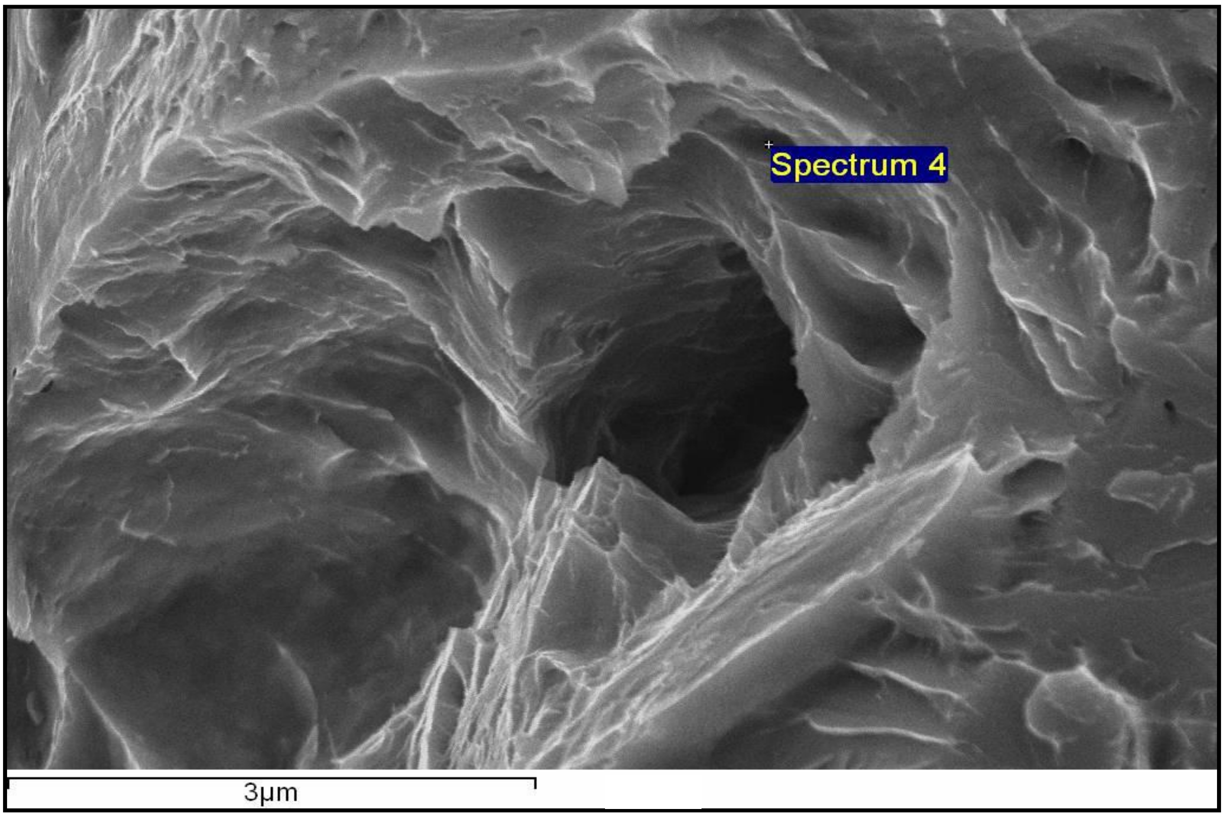


Figure 4.22 SEM Fractographic for Alloy 2 at Room Temperature after heat treatment

It is clear that the fracture surface shows complete facets with some impeded carbides, so as to the heat treatment has not any beneficial effect on the mechanical properties. These facets refer to the low values of impact toughness at room temperature after the heat treatment cycle.

A qualitative XRD analysis was applied to the impeded carbides as illustrated in figure 4.23 and tabulated in table 4.9



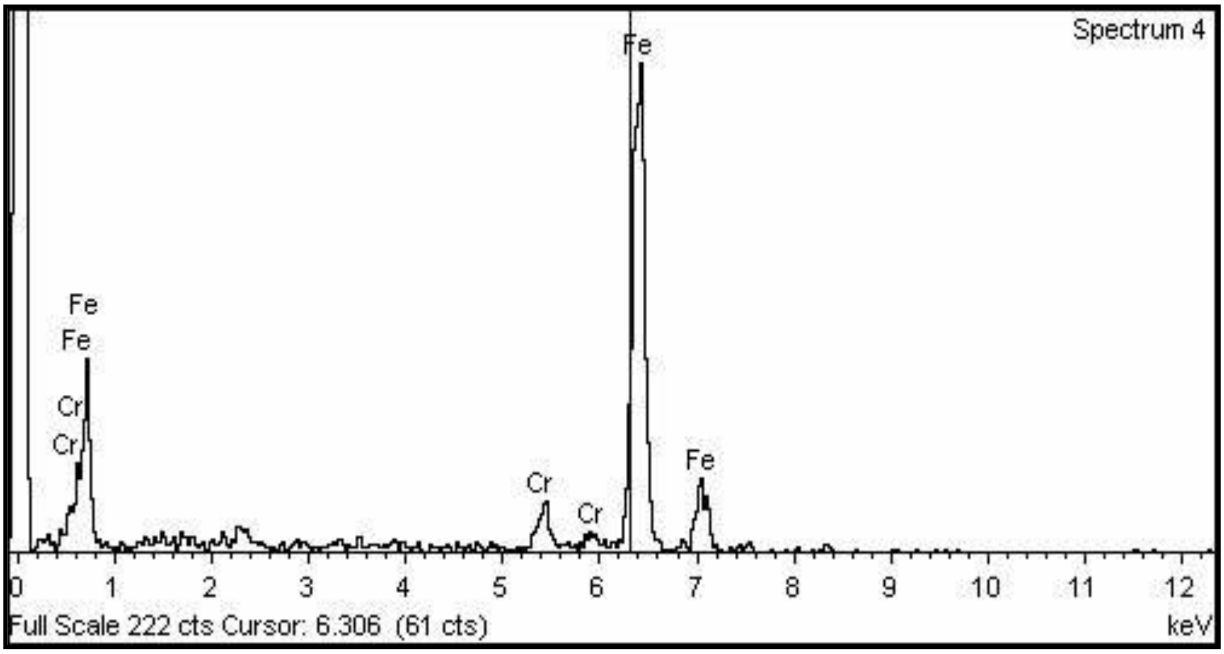


Figure 4.23 XRD for Alloy 2 after heat treatment

Table 4.9 XRD Analysis for Alloy 2 before heat treatment

Element	Cr	Fe
Weight%	4.75	95.25
Atomic%	5.09	94.91

The common carbides found by the XRD analysis are Chromium carbide and iron carbide.

Figure 4.24 gives illustration for the heat treatment fracture surface at -50 °C.

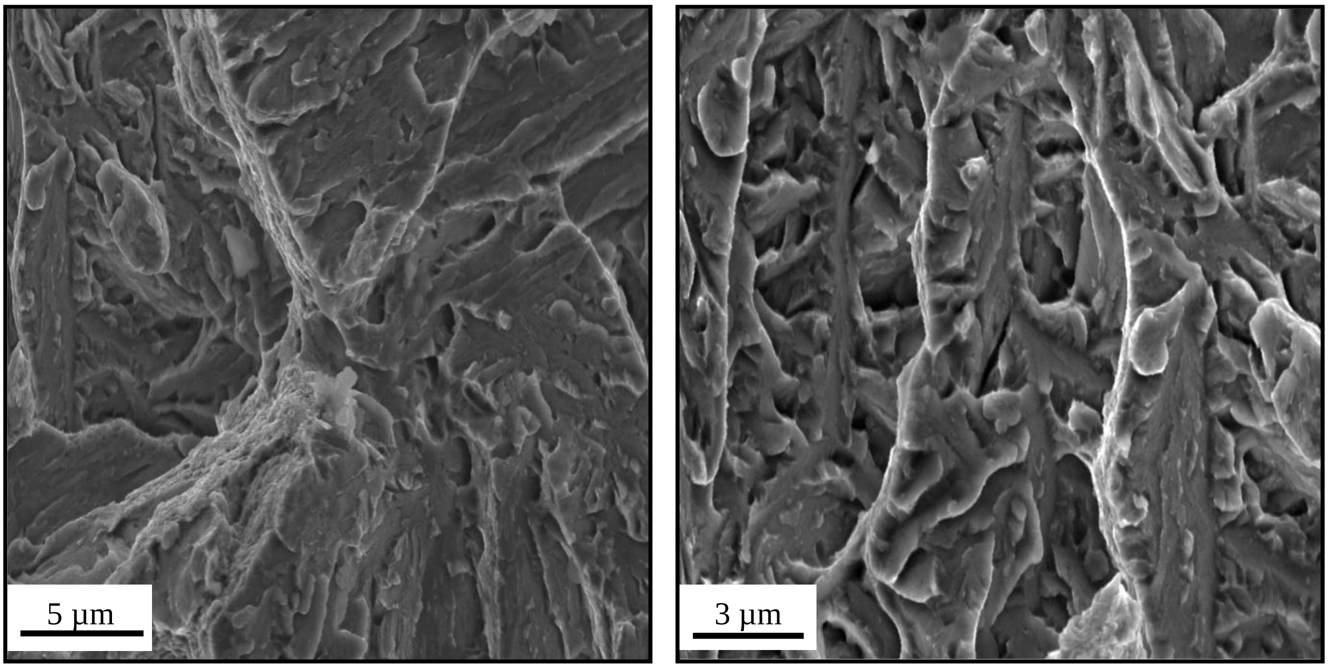


Figure 4.24 SEM Fractographic for Alloy 2 at -50 °C after heat treatment

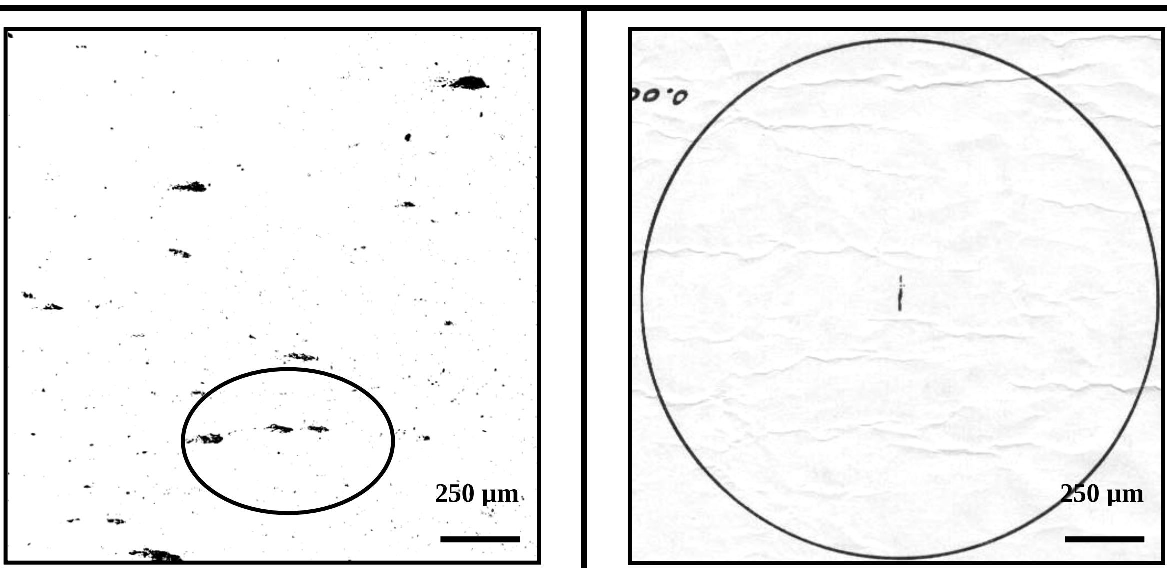
Figure 4.24 illustrates that the fracture surface is consists of small facets which leads to easy fracture (low toughness) at low temperature.

4.3. Investigation of alloy 3

Niobium is working as a grain refiner even at low concentration in alloy. Alloy 3 was designed to contain 0.06 % Niobium in order to increase the strength and toughness.

4.3.1. Non metallic inclusions in alloy 3.

Figure 4.25 represents the non metallic inclusions of investigated alloy 3.



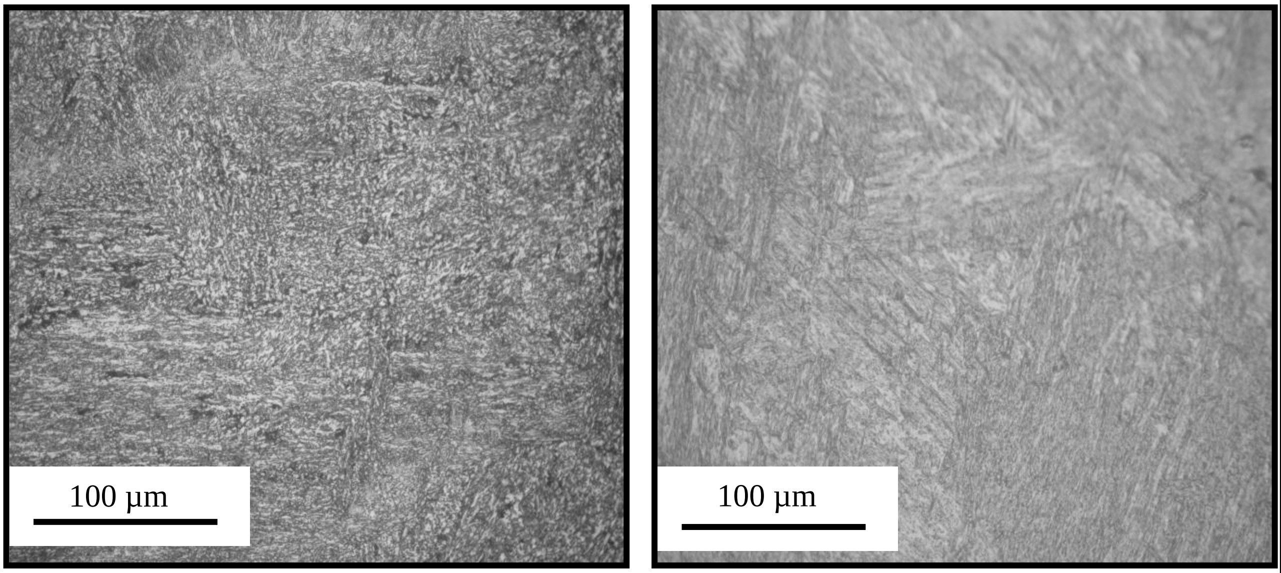
a) Actual non metallic inclusions for alloy 3	b) Standard Silicate type of non metallic inclusion according to ASTM

Figure 4.25 Actual non metallic inclusion comparisons for alloy 3 with standard Silicate.

Alloy 3 contains non metallic inclusion mixture of silicates and oxides as illustrated in figure 4.25. It is noticed that the amount of non metallic inclusions is higher than that found in the previous alloys, as it was the last portion of the 100 Kg melt.

4.3.2. Microstructure investigation for alloy 3.

Figure 4.26 represents both the as cast and heat treated microstructure of alloy 3.



(a) As cast microstructure (b) Heat treated microstructure

Figure 4.26 Microstructure for alloy 3

The refining effect of Niobium is reflected on the as cast microstructure where it is composed of fine martensite structure. The heat treatment is severely coarse the martensite structure which would negatively affect the mechanical properties [11-24-25-26].

4.3.3. Mechanical properties evaluation for alloy 3

4.3.3.1. Tensile test results for alloy 3

Tensile test was used as a tool to evaluate the effect of heat treatment. Table 4.10 illustrates the tensile results for alloy 3.

Table 4.10 Actual tensile test properties for alloy 3

Alloy	Condition	Specimen number	Tensile mechanical properties	
			UTS [MPa]	Mean UTS [MPa]
Alloy 3	As cast	1	938.85	930.9
		2	922.37	
		3	931.48	
	Heat treated	1	848.67	862.6
		2	882.6	
		3	856.53	

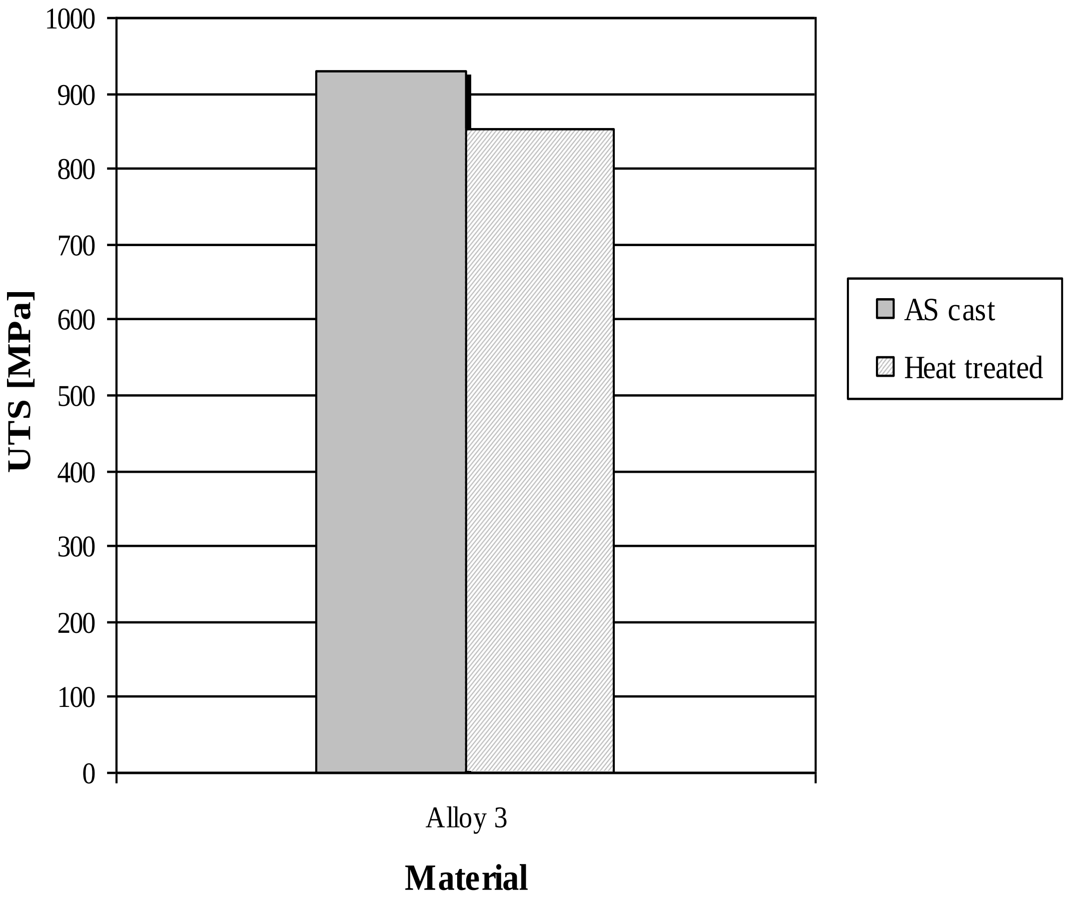


Figure 4.27 Tensile test results for alloy 3

The ultimate tensile strength of the as cast state is higher that the heat treated which was expected from the microstructure previously investigated, consequently, the applied heat treatment was not suitable for alloys containing Niobium micro-alloying.

4.3.3.2. Impact test results for alloy 3

Impact toughness at different ambient temperatures has been measured for both states (as cast-heat treated) and tabulated in table 4.11 and represents in figure 4.28.

Table 4.11 Impact properties for alloy 3

Condition	Test temperature [^{o}C]	Mean energy value [J]
As Cast	Room temp. (27 ^{o}C)	11.3
	-20	9.7
	-40	8.1
	-60	6.6
	-73	5.4
Heat Treated	Room temp. (27 ^{o}C)	9.2
	-20	8.3
	-40	6.5
	-60	5.7
	-73	5.2

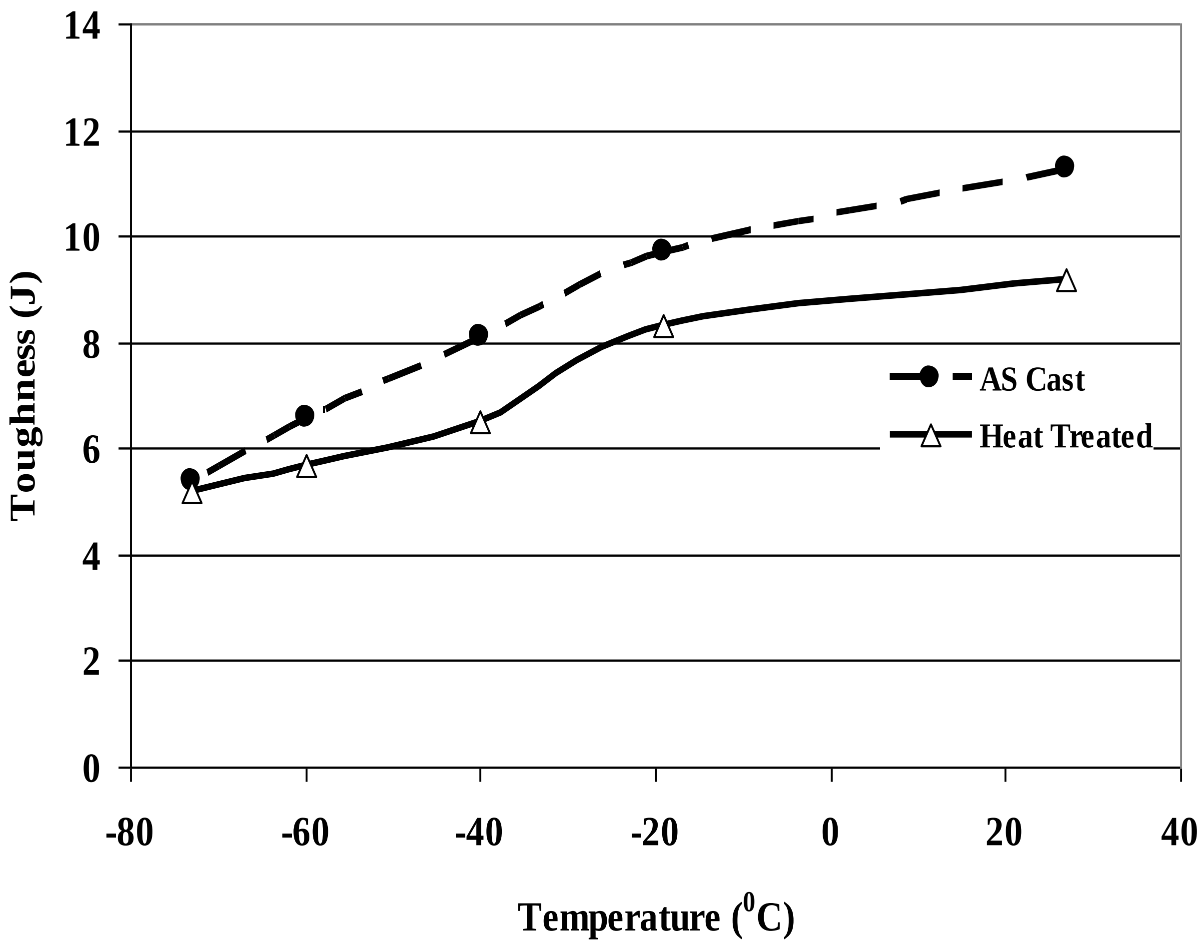


Figure 4.28 Impact toughness results for alloy 3 before and after heat treatment

It is noticed that the steel alloy 3 loss some toughness as it is subjected to deeply low temperature. Many previous works confirms what have been obtained [9-13].

On the other hand, toughness behavior of the heat treated alloy 3 is lower than that of the as cast as a result of unsuitable heat treatment.