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Effect of Low Temperatures on Charpy Impact Toughness of Austempered Ductile Irons

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Impact properties of standard American Society for Testing Materials (ASTM) grades of austempered ductile iron (ADI) were evaluated at subzero temperatures in unnotched and V-notched conditions and compared with ferritic and pearlitic grades of ductile irons (DIs). It was determined that there is a decrease in impact toughness for all ADI grades when there is a decrease in content of retained austenite and a decrease in test temperature, from room temperature (RT) to −60 °C. However, the difference in impact toughness values was not so noticeable for low retained austenite containing grade 5 ADI at both room and subzero temperatures as it was for ADI grade 1. Furthermore, the difference in impact toughness values of V-notched specimens of ADI grades 1 and 5 tested at −40 °C was minimal. The impact behaviors of ADI grade 5 and ferritic DI were found to be more stable than those of ADI grades 1, 2, 3, and 4 and pearlitic DI when the testing temperature was decreased. The impact toughness of ferritic DI was higher than that of ADI grades 1 and 2 at both −40 °C and −60 °C. The impact properties of ADI grades 4 and 5 were found to be higher than that of pearlitic DI at both −40 °C and −60 °C. The scanning electron microscopy (SEM) study of fracture surfaces revealed mixed ductile and quasicleavage rupture morphology types in all ADI samples tested at both −40 °C and −60 °C. With decreasing content of retained austenite and ductility, the number of quasicleavage facets increased from ADI grade 1-5. It was also found that fracture morphology of ADI did not experience significant changes when the testing temperature decreased. Evaluation of the bending angle was used to support impact-testing data. Designers and users of ADI castings may use the data developed in this research as a reference.

Keywords ausferrite, austempered ductile iron (ADI), austempering, ferritic ductile iron, pearlitic ductile iron, retained austenite

1. Introduction

Austempered ductile iron (ADI) is a relatively new industrial material that has excellent ductility, strength, and toughness properties.[1] ADI exhibits more than twice the strength for a given level of ductility compared with conventional ductile irons (DIs).[2] Although the level of its mechanical properties is almost the same as in high-alloy steels, the production cost of ADI is significantly lower.[3]

According to a recent study,[4] ADI is a heat-treated nodular cast iron with a unique microstructure that consists of stabilized high carbon (C) austenite and acicular ferrite with graphite nodules dispersed in it. Ausferrite is a standard name of ADI microstructure as per American Society for Testing Materials (ASTM) A644-92.

To obtain its unique properties, the as-cast ductile iron is subjected to the special austempering heat treatment cycle (Fig. 1),[5] which consists of heating and holding a casting in the temperature range of 840-900 °C followed by quenching in a molten salt bath and subsequently cooling to the austempering temperature, which varies from 220-400 °C depending upon the desirable ADI grade. After holding a casting at the austempering temperature for about 2-4 h, it is air-cooled to room temperature (RT).[6] After the austempering, the microstructure consists of ausferrite and graphite nodules. The typical microstructures of DI before and after austempering are shown in Fig. 2.

There is a risk of bainitic transformation in ADI microstructure if a casting is held at the austempering temperature longer than necessary. In this case, austenite is not able to hold C in the solution any longer and the carbides nucleation begins, with all austenite transforming into ferrite and almost all carbon forming bainitic carbides. As a result of these detrimental transformations in ADI, the microstructure of iron becomes fully bainitic.

A literature review has revealed the fact that the published data on mechanical properties, particularly on impact toughness, are insufficient or related primarily to austempering pro-

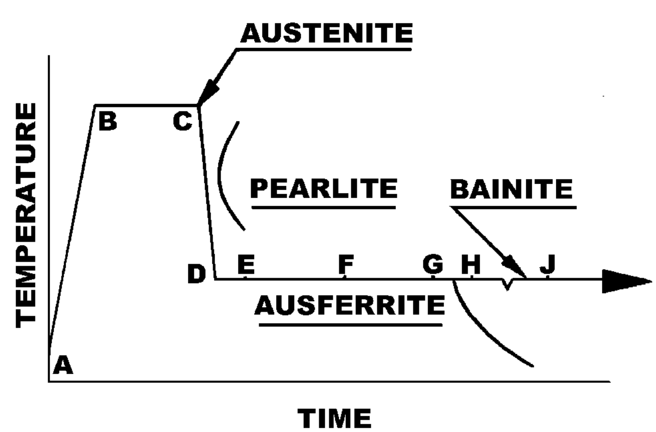

Fig. 1 Schematic diagram of the austempering heat treatment cycle[5]

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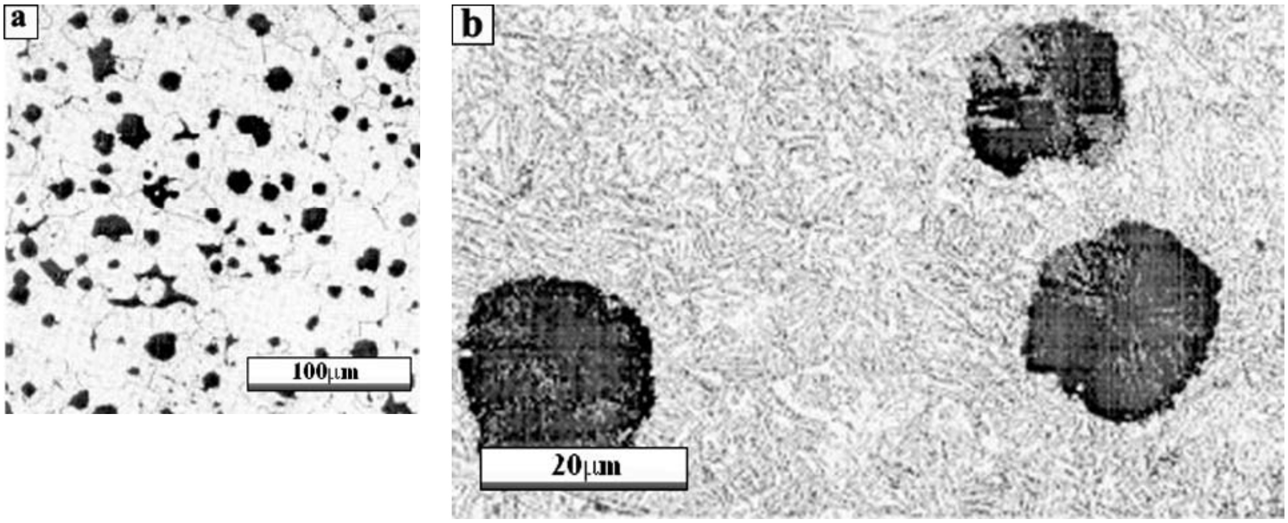

Fig. 2 **(a)** The microstructure of as-cast DI, 100X, 2% Nital; **(b)** The microstructure of ADI austenitized at 870 °C for 3 h and austempered at 385 °C for 2 h, 500×, 2% Nital[7]

cess parameters rather than to the standard ADI grades. This can be confusing to the designer who may not have a metallurgical background adequate to interpret the published information with confidence. All previous research had the intention of specifying the influence of low testing temperatures on yield strength and tensile strength.

According to the literature,[7] accurate data regarding ductile-to-brittle transition temperatures and consequently the impact behavior of ADI at low temperatures (LT) (−40 °C and −60 °C) are not available. British scientists[8,9] conducted some studies to determine impact properties of ADI at LT, but those experiments were performed for the British Cast Iron Research Association (BCIRA) tentative ADI grades that are different from ASTM grades.

Considering current and potential application areas of ADI such as in automotive industry (transmission and suspension), earth-moving equipment, tractors, etc., the knowledge of impact behavior of ADI at subzero temperatures is very useful.

The objective of this research was to study the impact properties of ASTM standard grades of ADI at low testing temperatures of −40 °C and −60 °C and to compare them with those of ferritic DI grade 60-40-18 and pearlitic DI grade 100-70-03, which were also tested at the same temperatures. Standard unnotched Charpy impact testing specimens (ASTM E23-96) were produced from ASTM A897-90 ADI grades 125/80/10, 150/100/7, 175/125/4, 200/155/1, and 230/185/- to achieve the goals of the research study. For simplicity, those grades will be further referred as grades 1, 2, 3, 4, and 5, respectively. V-notched samples were produced from only ADI grades 1 and 5, which represent maximum and minimum ductility, respectively. The samples were then tested at −40 °C and −60 °C in accordance with ASTM E23-96 procedures. Although this standard specifies impact testing of notched specimens for metallic materials, it was decided to use both unnotched and V-notched samples because of the low impact value of V-notched specimens and the difficulty of noticing any slight changes in impact toughness.

Table 1 Austempering Heat Treatment Parameters

ADI Grade	Austenitizing Temp, °C (°F)	Holding Time, h	Austempering Temp, °C (°F)	Holding Time, h
1	885 (1625)	1.67	357 (675)	1.5
2	885 (1625)	4	329 (625)	2
3	885 (1625)	4	313 (560)	2.5
4	885 (1625)	4	293 (560)	2.5
5	885 (1625)	2	271 (520)	3.5

2. Experimental Procedures

2.1 Production of the Test Material

Base DI for the austempering was produced in a 300 lb medium frequency coreless induction furnace from a charge mix consisting of Sorel pig iron, AISI 1010 steel punchings, and FeSi 75. The flow-through technique was used to treat the melt with 2% of FeSiMg master alloy, containing 6.2% Mg, at 1490 °C (2750 °F). To post-inoculate the iron, 0.5% of an inoculation grade FeSi 75 was placed into the reaction chamber of the treatment unit along with the magnesium treatment master alloy. The final chemical composition of DI was as follows (wt.%): 3.60-3.65 C, 2.50-2.60 Si, 0.25-0.30 Mn, and 0.04-0.06 Mg.

The iron was cast in no-bake sand molds in the shape of 8″ × 8″ × 0.5″ plates. The as-cast microstructure was pearlitic-ferritic, with a pearlite content of approximately 60% and the graphite nodularity of about 90%.

As-cast plates were austempered at a specialized austempering heat treatment facility with heat treatment parameters presented in Table 1. Upon the completion of austempering, the plates were removed from the salt bath and washed with water to remove any adhering salt from the austempering process. Finally, the plates were air-cooled to RT. A few plates were then used for the production of materials for test results comparison (i.e., ferritic DI grade 60-40-18 and pearlitic DI grade

Table 2 Results of ADI Testing at Room Temperature

Mechanical Property	ADI Grade 1	ADI Grade 2	ADI Grade 3	ADI Grade 4	ADI Grade 5
Tensile Strength, MPa (ksi)	1089 (158)	1069 (155)	1345 (195)	1427 (207)	1469 (213)
Yield Strength, MPa (ksi)	827 (120)	841 (122)	1041 (151)	1200 (174)	1267 (184)
Elongation, %	11	10.5	7	3.5	3
Reduced Area, %	…	…	3.5	4	…
Hardness, HB (HRC) (a)	292 (31.3)	301 (32.5)	357 (38.5)	388 (41.5)	404 (43)
Impact Toughness, J (ft-lb)	127 (94)	118 (86.5)	96 (71)	73 (54)	38 (27.5)

(a) Rockwell Hardness (HRC) values converted from Brinell Hardness (HB).

100-70-03). Next, as-cast samples were annealed at the temperature of 930 ± 10 °C (1700 ± 10 °F) for 3 h and then cooled in the furnace to RT to produce ferritic DI grade 60-40-18. Pearlitic DI grade 100-70-03 samples were produced by normalizing at 930 ± 10 °C (1700 ± 10 °F) for 3 h and then removed from the furnace and air-cooled to RT.

The metallic matrix after annealing was almost 100% ferritic, whereas after normalizing, the matrix was fully pearlitic and contained not more than 3-5% ferrite.

2.2 Determination of Mechanical Properties at Room Temperature

Mechanical properties at RT were evaluated under the guidelines of ASTM A897 Standard Specification for ADI Castings. Table 2 contains the actual mechanical properties of ADI used in this research.

2.3 Impact Toughness Testing at −40 °C and −60 °C

Charpy impact toughness testing was completed in accordance with ASTM E-23 standard testing procedure. Cast plates were cut and machined to provide standard rectangular unnotched samples with dimensions 10 mm × 10 mm × 55 mm. All samples were machined using coolant at all stages, including polishing to avoid phase transformation in the solid state.

The current research project consisted of two stages. In the first series, five unnotched specimens of each ADI grade were tested at temperatures of −40 °C and −60 °C along with five ferritic DI grade 60-40-18 and five pearlitic DI grade 100-70-03 comparative samples. Five V-notched samples of each ADI grade 1 and ADI grade 5 were tested in the second series at −40 °C. Testing temperatures of −40 °C and −60 °C were obtained using the cooling medium consisting of dry ice and denatured alcohol. A tank eight inches in diameter and eight inches high was used as the container for cooling specimens down to the aforementioned temperatures. Samples were held in a screen 4 in. off the bottom of the tank and were soaked for at least 30 min. All samples were tested within 5 s after their removal from the cooling medium.

Fracture surface morphology was analyzed using visual, stereoscopic microscope observations, and scanning electron microscope (SEM) techniques. The retained austenite content in the ADI microstructure was determined using the quantitative x-ray diffraction (XRD) method.

As an extra tool for fracture mechanism analysis, an additional methodology was used in this study. This methodology includes the determination of a specimen's degree of deformation, which according to the literature,[8] may be a guide to whether the rupture occurred during a ductile or brittle mode. The bending angle of specimens was determined to support results obtained through impact testing. A sample was projected on a plain surface to obtain the replica of its deformed surface and the bending angle was measured using a bevel gauge.

3. Results and Discussion

3.1 Retained Austenite in Relation to Mechanical Properties of ADI

The results of the retained austenite content evaluation in the microstructure of studied ADI samples are presented in Fig. 3. ADI grade 1, which had the lowest tensile strength and the highest elongation, contained about 40% of retained austenite in the microstructure. The retained austenite content in microstructure of ADI grade 5, which had the highest tensile properties but zero ductility, was only 4.8%. The rest of the metallic matrix was found to be acicular ferrite. The difference in the retained austenite composition was stipulated to be due to the difference in the austempering parameters. High-temperature austempering led to a higher amount of retained austenite in the microstructure of ADI. These data are in good agreement with a study reported previously,[10] which stated that retained austenite greatly affects the mechanical properties of ADI.

3.2 Impact Toughness Testing

The results of impact toughness testing of all ADI samples at room and subzero temperatures as arithmetic means are presented in Fig. 4. According to the obtained data, there is a general trend of decreasing impact toughness for all ADI grades as the testing temperature was reduced. It is clear that when the retained austenite content was reduced, the impact toughness of ADI gradually decreased. However, the difference in the impact toughness values was not so noticeable for low retained austenite containing ADI grade 5 at both room and subzero temperatures as it was for ADI grade 1. As expected, the difference in impact toughness values of V-notched experimental specimens tested at −40 °C was also minor: namely, 10.5–11 J for ADI grade 1 and 9–10 J for ADI grade 5. Figure 5 shows the mean values of unnotched impact values of ADI in

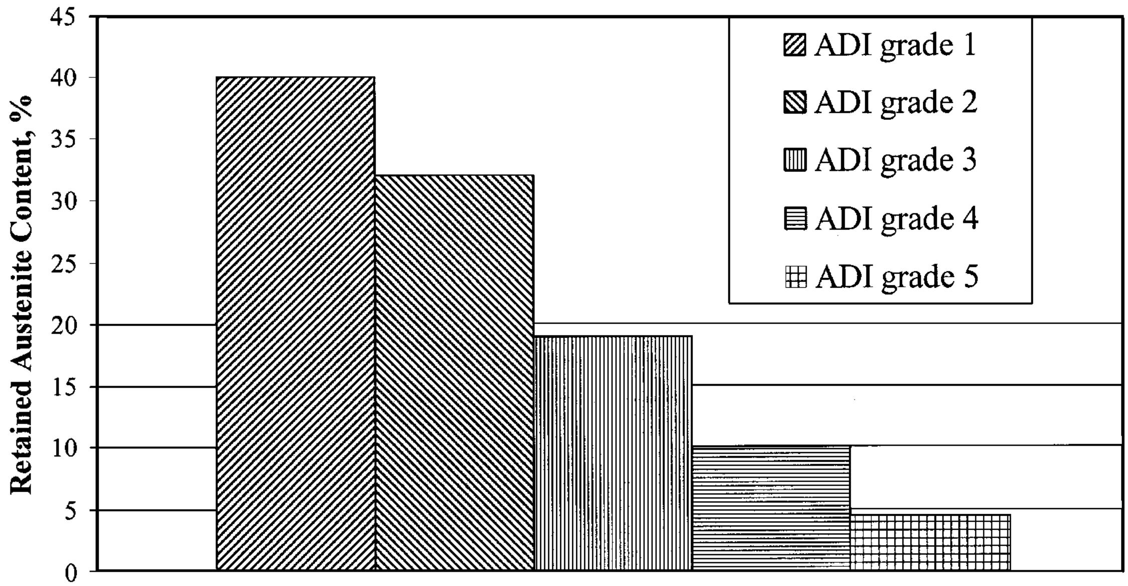

Fig. 3 Retained austenite content in ASTM standard ADI grades

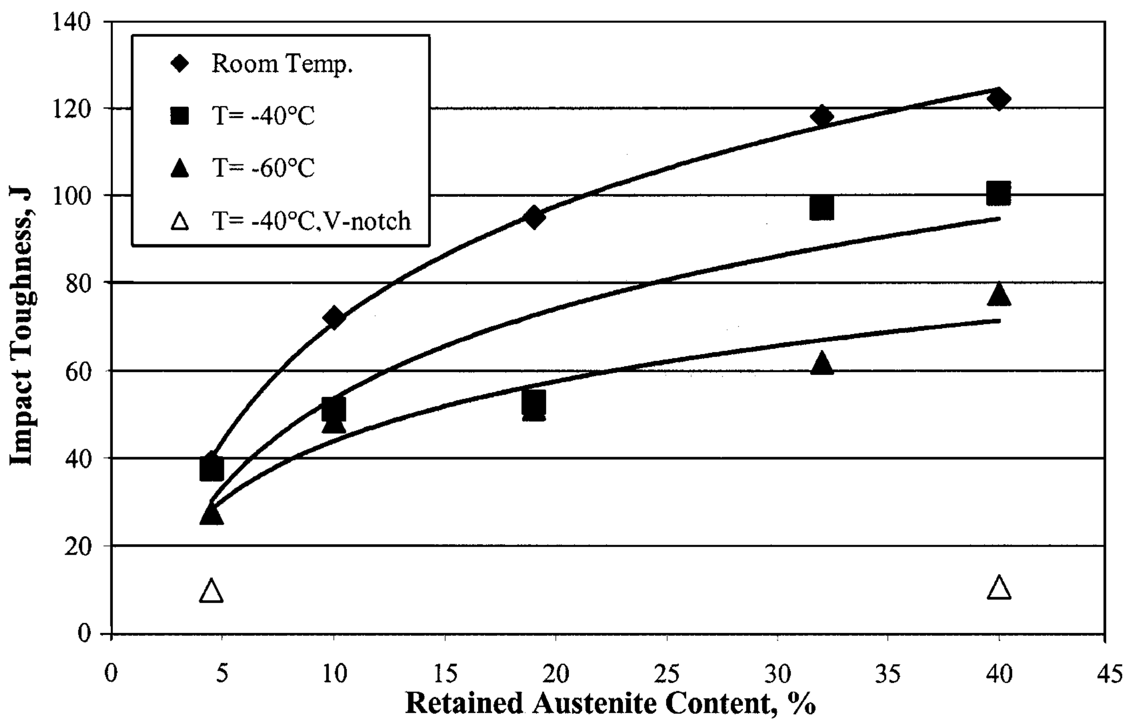

Fig. 4 The impact toughness of ADI samples tested at temperatures of −40 °C and −60 °C as a function of retained austenite content. The data are given in comparison with RT impact tests.

comparison with ferritic DI grade 60-40-18 and pearlitic DI grade 100-70-03 tested at room and subzero temperatures. As can be seen, with the reduction of the testing temperature, the impact toughness of all tested materials decreased. The impact toughness of ferritic DI grade 60-40-18 was higher than that of all ADI grades at room and subzero temperatures as well. Pearlitic DI grade 100-70-03 had the lowest impact toughness among all tested materials: namely, 33 J for iron tested at −40 °C and 21 J for iron tested at −60 °C. However, when these two materials were compared, the impact behavior of ferritic DI grade 60-40-18 appeared to be more stable when the testing temperature decreased, and the impact toughness of ferritic DI grade 60-40-18 was approximately 3.1 times higher than that of pearlitic DI grade 100-70-03 tested at RT. Impact toughness of ferritic DI was found to be about 3.8 times greater than that of pearlitic DI tested at −40 °C and about 5.8 times greater at

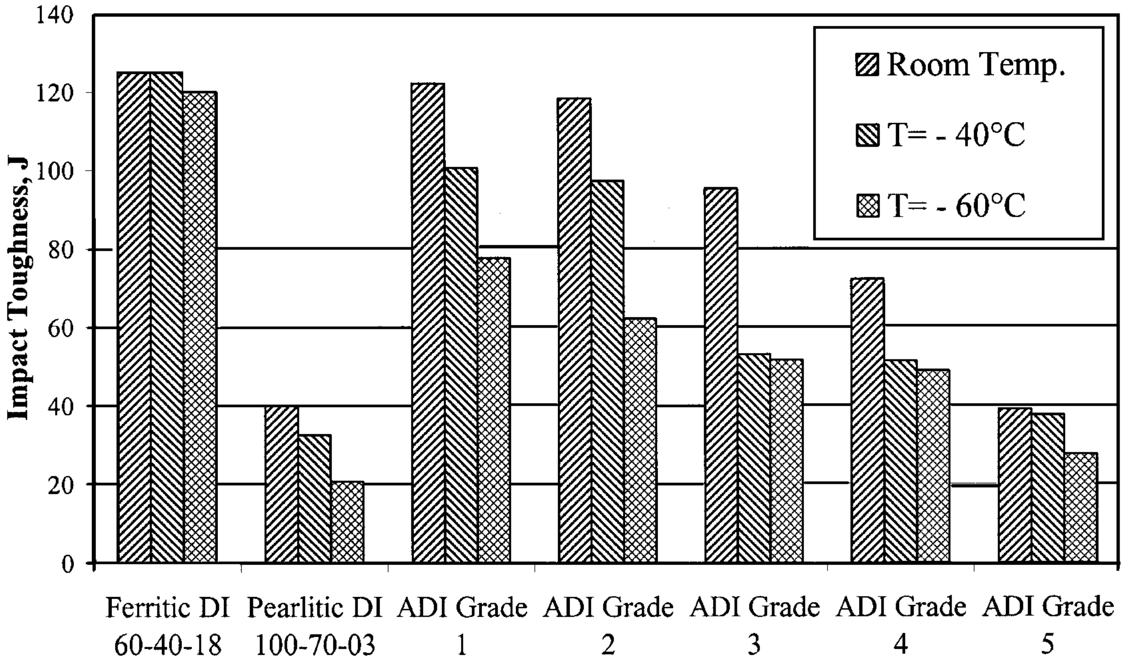

Fig. 5 The unnotched impact values of ADI in comparison with ferritic and pearlitic DIs, tested at room and subzero temperatures

–60 °C. Comparison of ferritic DI grade 60-40-18 and ADI grades 1 and 2 with close levels of impact properties showed that the impact toughness of ferritic DI was only 5% and 25% higher than that of ADI grades 1 and 2 tested at RT and at –40 °C, respectively. When tested at –60 °C, ferritic DI had impact toughness values approximately 1.5 times greater than that of ADI grade 1 and approximately 2 times higher than that of ADI grade 2. Impact properties of pearlitic DI grade 100-70-03 and ADI grade 5 were close—pearlitic DI had impact toughness approximately 5% higher than that of ADI grade 5 tested at RT. However, impact toughness of ADI grade 5 was approximately 10% and 25% higher than that of pearlitic DI tested at –40 °C and –60 °C, respectively.

3.3 Fracture Analysis

Visual and stereomicroscope observations of ADI samples tested at subzero temperatures showed that all samples had light gray fractures regardless of their impact values, and it was impossible to differentiate grades of ADI according to visual observation of their fracture appearance only. The fracture of ADI occurred through the grains and contained only a few nodules and was similar to those of DI grade 100-70-03 with pearlitic metallic matrix. Annealed ferritic DI grade 60-40-18 had a clearly ductile dark gray color fracture, with a number of glistery graphite inclusions as a result of fracture occurrence along non-reflective grain boundaries.

Fracture surfaces of some samples were evaluated using scanning electron microscopy (SEM). Relatively low magnification of 500× was used for fractographs because the fracture surfaces were not flat enough to obtain good focus at high magnifications. As can be seen in Fig. 6, 7, and 8, the predominant fracture mechanism was of the cleavage (brittle) fracture mode and was evidenced by faceted shiny fracture surfaces. Specimens tested at lower temperatures (–60 °C) started to show voids forming around the graphite nodules. It was found that with increasing tensile properties of ADI, the number of quasicleavage facets on the fracture surface increased from grade 1-5. Decreasing the testing temperature from –40–60 °C did not have significant influence on fracture morphology of ADI.

The ferritic DI grade showed a significant ductile fracture mechanism as evidenced by the dominant riverlines exhibited in Fig. 9. Ductile fracture occurred as a result of void formation at the interface between the two phases, shown clearly in Fig. 9. Such void formation was followed by void growth and led to fracture. The observation of SEM fractographs of pearlitic DI grade 100-70-03 tested at –40 °C and –60 °C showed that these samples had mixed ductile and quasicleavage type of rupture morphology that was similar to that of ADI samples.

3.4 The Analysis of Sample Deformation

The deformation of samples before their rupture was used as another measure for quantifying impact fracture. Measuring the bending angle of a sample before fracture was used for this purpose. Figure 10 presents the results of evaluation of these parameters for materials tested at –40 °C. Ferritic DI grade 60-40-18 had the highest bending angle value of about 16.5 degrees. All tested ADI samples, except for those of ADI grade 5, ruptured in mixed ductile-brittle mode, with the bending angle gradually decreasing from ADI grade 1 to ADI grade 4. ADI grades 1 and 2 had bending angle values close to that of DI with ferritic metallic matrix, of approximately 12.9 and 11.5°, respectively. The bending angles of ADI grades 3 and 4 were approximately 8 and 4.5°, respectively. ADI grade 5 samples had bending angles of about 2.5°, which was close to

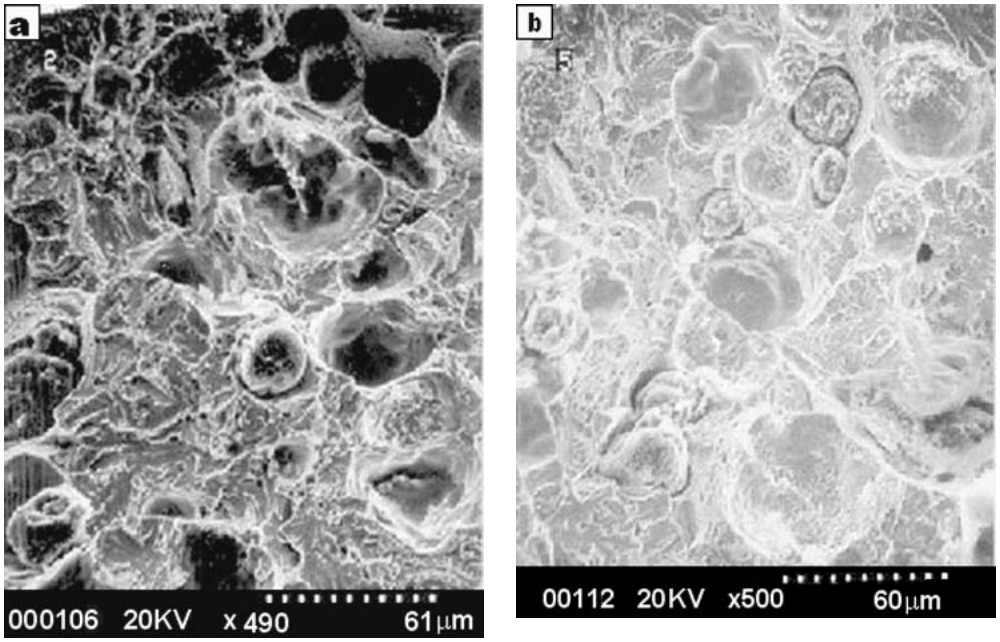

Fig. 6 SEM fractographs of ASTM ADI grade 1 tested at −40 °C **(a)** and −60 °C **(b)**, magnification 500×

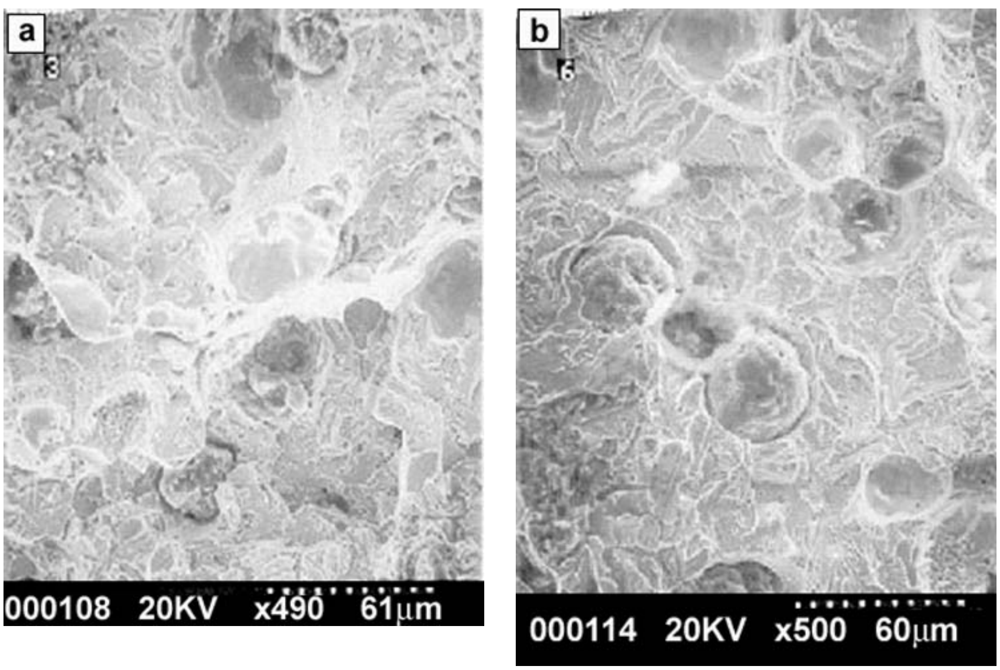

Fig. 7 SEM fractographs of ASTM ADI grade 3 tested at −40 °C **(a)** and −60 °C **(b)**, magnification 500×

the bending angle of pearlitic DI grade 100-70-03 sample (2°), and it would be possible to conclude that in both cases predominantly brittle fracture occurred.

The results obtained from the bending angle determination in Fig. 10 show trends similar to those shown by impact energy in Fig. 5. However, it does not seem practical to use bending angle measurements to conclusively determine the impact properties of materials.

4. Conclusions

In conclusion, the results obtained during this study showed that the impact toughness values of all ADI grades decreased with the reduction of the testing temperature from RT to −60 °C and with the reduction of retained austenite content. However, the gap between impact toughness values at room and

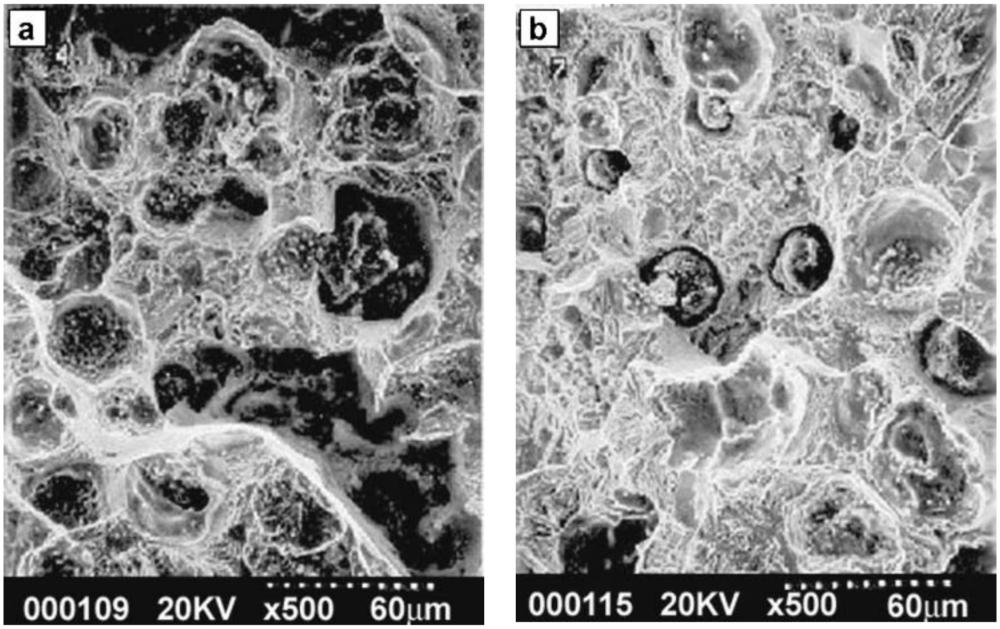

Fig. 8 SEM fractographs of ASTM ADI grade 5 tested at −40 °C (**a**) and −60 °C (**b**), magnification 500×

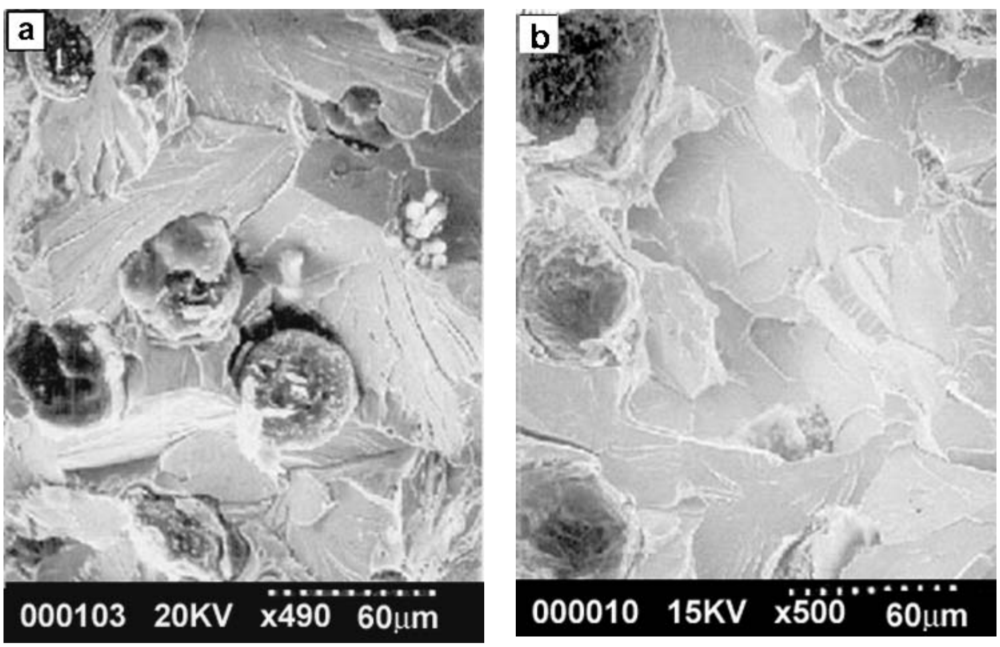

Fig. 9 SEM fractographs of ferritic DI grade 60-40-18 tested at −40 °C (**a**) and −60 °C (**b**), magnification 490×

subzero temperatures was wider for high retained austenite containing ADI grade 1 than for ADI grade 5. As expected, the difference in the impact toughness values of V-notched experimental specimens tested at −40 °C was minimal.

The impact behavior of ADI grade 5 and ferritic DI grade 60-40-18 was more stable than those of ADI grades 1, 2, 3, and 4 and pearlitic DI grade 100-70-03 when the testing temperature was decreased. The impact toughness of ferritic DI grade 60-40-18 was greater than that of ADI grades 1 and 2 at both −40 °C and −60 °C. ADI grades 4 and 5 had the level of impact properties higher than that of pearlitic DI grade 100-70-03 at both −40 °C and −60 °C.

Visual stereoscopic observations of ADI samples showed that all samples had fractures of light gray color, even though there was a noticeable difference in impact toughness values. The SEM study of fractured surfaces revealed that all ADI samples tested at both −40 °C and −60 °C had mixed ductile and quasicleavage type of rupture morphology. With decreasing retained austenite content and ductility, the number of quasicleavage facets increased from ADI grade 1-5.

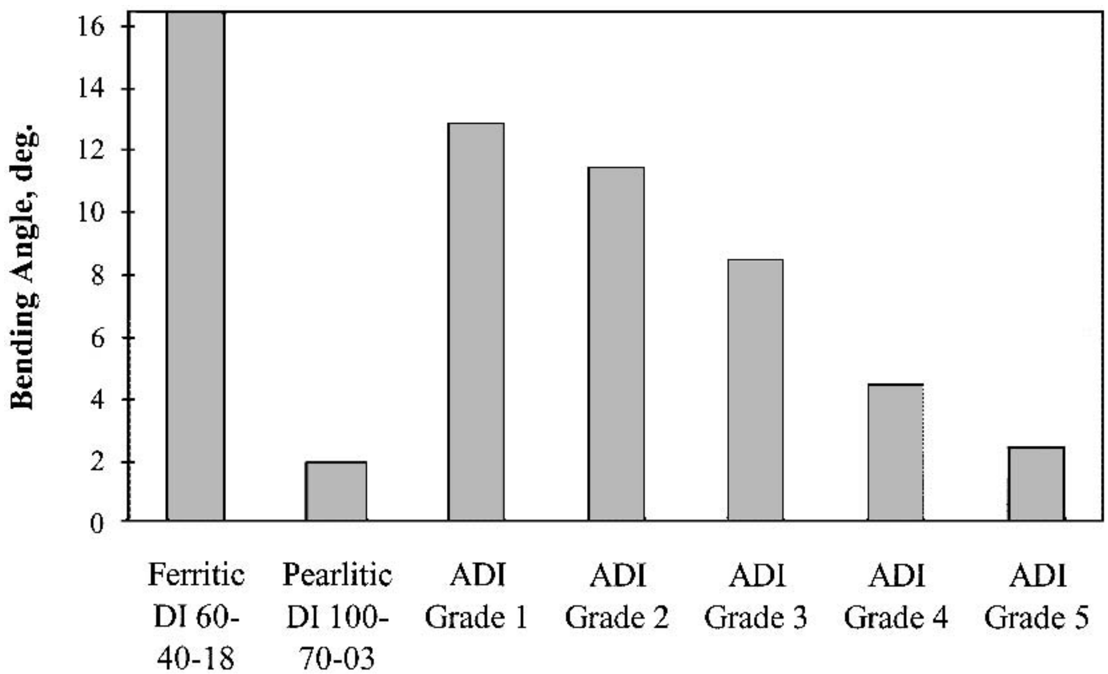

Fig. 10 Bending angle of unnotched Charpy impact specimens tested at −40 °C

Testing temperatures applied in this research did not have significant influence on the fracture morphology of ADI.

Bending angle measurements showed trends similar to those shown by impact energy. Hence, impact energy is a simple and direct measure of the quality and fracture resistance of this material.

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