Tensile Properties of Austempered Ductile Iron under Thermomechanical Treatment

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A new processing method was investigated for improving the strength and elongation of austempered ductile iron (ADI) by grain refinement of parent austenite using thermomechanical treatment. The material was deformed at the austenitization temperature by single and multipass rolling before the austempering treatment. The effects of the amount of deformation, austenitization temperature, austempering temperatures, reaustenitization, and secondary deformation on the tensile properties were studied. The properties obtained using the method were compared with those of the ASTM standards. The effect of deformation on the graphite shape was also studied. Tensile strength/yield strength/elongation values were found to increase with increasing austenite deformation up to 40% and then to start decreasing. Tensile strength/yield strength and elongation values of 1700 MPa/1300 MPa/5% and 1350 MPa/920 MPa/15% can be achieved with this method in the ranges of variables studied.

Keywords austemper, ductile iron, heat treatment, thermomechanical forming

1. Introduction

Austempered ductile iron (ADI) offers the best combination of low cost, design flexibility, machinability, strength-to-weight ratio, toughness, wear resistance, and fatigue resistance and also allows the manufacturer to obtain a wide range of properties in a component. The combination of properties achievable with ADI makes it competitive with steel in many applications. Austempered ductile iron is east like any other member of the ductile iron family, offering the production advantages of conventional ductile iron, and then austempered to obtain mechanical properties that are superior to all cast irons and many cast steels.[1]

Austempering treatment generally consists of (a) fully austenitizing the iron at suitable austenitizing temperature, (b) quenching to an austempering temperature and holding it for an appropriate length of time for the isothermal transformation, and (c) air cooling to room temperature. Quenching from austenitizing temperature to austempering temperature should be rapid enough to avoid the formation of ferrite and pearlite in order to maximize toughness and ductility. The austempering reaction in ADI occurs in two stages. In the first stage, austenite matrix transforms to a mixture of acicular ferrite and carbon-enriched stabilized austenite, termed appropriately as ausferrite. The second stage consists of decomposition of the carbon-enriched austenite to a ferrite-carbide aggregate. The best combination of properties in ADI is obtained when austempering treatment is stopped when the first stage is nearly complete, but the transformation has not progressed well into the second stage. The austempering transformation in ADI differs from the analogous reaction in steels where austenite decomposes uniformly into the ferrite carbide aggregate called bainite in a relatively shorter period of time.[2]

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The mechanical properties of ADI depend on the relative amounts of acicular ferrite and stabilized austenite and on the morphology of the former. These in turn are determined by the austempering temperature and time. Higher austempering temperatures produce coarser matrix microstructure consisting of a larger volume fraction of stabilized austenite and a lesser amount of ferrite, giving the material lower strength and higher ductility. Austempering at lower temperatures, conversely, produces a microstructure comprised of a larger volume fraction of acicular ferrite and lower amounts of stabilized austenite. This material has higher strength and lower ductility. The acicular ferrite morphology changes from feathery at higher temperatures to more needle shaped at lower temperatures, which also contributes to the reduction in ductility. The phase morphology is finer, and the size of the austenite and ferrite platelets is smaller at lower temperatures.[3]

If the austempering time is too short, the first stage of the austempering reaction will be incomplete and the unreacted parent austenite will transform to martensite, resulting in poor toughness and ductility. Conversely, prolonged holding at austempering temperature will result in austenite enrichment to the point where it becomes less stable, and the second stage of the reaction occurs, resulting in more stable ferrite and carbide phases. This results in loss of toughness and ductility.

The mechanical properties of ADI also depend on the austenitization temperature. The matrix carbon content in ADI is not constant as in steel, but is a function of austenitizing temperature and composition of the iron, especially its silicon and manganese content. If the temperature and time are too low, austenitization may be incomplete, or the carbon content may be lower than the saturation amount, resulting in either low carbon in the reacted austenite or longer austempering time. In the former case, austenite in the resulting ausferrite will not be stabilized, and martensitic transformation is likely to occur. In the latter, the volume fraction of reacted austenite and the mechanical properties are lowered, and the production cost is increased. Conversely, high austenitizing temperatures increase the matrix carbon content, which delays and slows the austempering reaction.[4]

Another effect of austenitizing temperature and time that is not discussed in the treatment of conventional ADI, but which is

important for the present study, is the effect on austenite grain size. It is reported that in steels, prior microstructure and holding time have no effect on the grain size at temperatures below 900 °C, while grain coarsening does occur beyond 950 °C. The resulting austenite grain size at these temperatures depends on the temperature and holding time. High austenitizing temperature and time would thus defeat the objective of grain refinement. Hence, for effective grain refinement, austenitization temperature and time should be the minimum necessary for complete transformation to, and carbon saturation of, austenite.

2. Thermomechanical Treatment of Austempered Ductile Iron

While ADI has an excellent combination of mechanical properties, certain characteristics impose limitations on its wider usage. The lower elongations in high strength ADI (such as grade 1600/1300 of ASTM A 897M) restrict the widespread use of ADI in applications requiring higher toughness. These high tensile and yield strengths are obtained by austempering at lower temperatures, but at the cost of ductility. In order to achieve a combination of higher tensile strength along with high ductility, it is necessary to investigate new methods of processing ADI. One of the methods is strengthening by grain refinement. The general relationship between yield strength, σ_Y, and grain diameter, D, is usually expressed by the Hall-Petch equation:[5,6]

$$\sigma_\gamma = \sigma_0 + kD^{1/2} \quad \text{(Eq 1)}$$

where σ_0 represents the resistance of the crystal lattice and k is the relative hardening contribution from the grain boundaries. The Hall-Petch equation has also been found to apply to other types of boundaries, such as ferrite/cementite in pearlite, martensite plates, and mechanical twins.[7] Fatigue strength, fracture strength, and ductile-brittle transition temperature are also improved with finer grain size.[8] Because ferrite phase nucleates on the austenite grain boundaries and grows into the austenite grain, the ferrite grain size is related to prior austenite grain size. Thus, refinement of ferrite is achieved mainly through refinement of austenite.[9] If the austenite grain size is reduced before the austempering transformation, the strengthening from the refined ferrite/stabilized austenite boundary will be in addition to the strengthening from prior austenite grain boundaries. Hence, one of the methods to improve the properties of ADI would be to refine the matrix microstructure consisting of feathery acicular ferrite and high volume fraction of stabilized austenite. In the case of steels, controlled rolling is employed to refine the structure in order to enhance both strength and toughness. The inherent ductility of ductile irons can be exploited by using a controlled rolling approach to enhance the strength along with ductility. This could be achieved by austempering a ductile iron with refined austenite at the austenitization stage, at appropriate temperature depending on the strength requirement. With finer prior austenite grains, the effects of segregation of alloying elements are also reduced. With this approach, the features and proportions of the phases rendering ductility will be maintained while higher tensile strengths can be achieved from the refined microstructure.

One of the methods of refining the matrix microstructure is thermomechanical processing, that is, mechanically working the austenite before austempering. Hot working will produce deformed and/or recrystallized austenite depending on the temperature of deformation and other hot working parameters involved, resulting in an increased number of sites for the nucleation of acicular ferrite during subsequent austempering. The deformation can be achieved by single or multiple pass hot rolling or hot pressing prior to austempering. Rolling at austenitization temperature will reduce the load per unit length on rolls and permit higher amounts of deformations compared to ausforming[10] at the austempering range of temperatures.

The possible changes occurring in the material during hot deformation process will be (a) flattening or pancaking of the austenite grains leading to work hardening, (b) softening due to dynamic recovery and recrystallization that occur during deformation, and (c) static recovery and recrystallization during the interpass period and subsequent grain growth. At first, the material is work hardened at the deformation temperature, due to the rolling stress. The austenite grains become compressed in the direction of the stress and elongated in the perpendicular direction. This elongation increases the surface area of austenite grains per unit volume and generates twin boundaries in the grains. Depending on the temperature, time, and percentage reduction, the material will undergo dynamic, static, or partial recovery followed by recrystallization.

During hot deformation, the graphite nodules also become deformed, introducing some degree of mechanical flattening and associated directionality in the properties. The strength and ductility of the iron are expected to be higher in the direction of deformation. The extent of graphite deformation and its effects on the anisotropy of mechanical properties will increase with an increase of nodule size. Hence, it is desirable to have a base iron with uniformly distributed small graphite nodules and high nodule counts so that the effects of graphite deformation are minimized, while advantages gained by the thermomechanical processing of the matrix are retained.

Graphite nodules in ductile iron in thermomechanical treatment can be regarded in the same way as the inclusions in rolling of steel. Then the effect of graphite on ductility depends on its shape and distribution and the stress system imposed during deformation. The initiation of fracture at the inclusion is dependent upon its deformation characteristics with respect to matrix.[11] Hence, when ductile iron is deformed, the following cases may arise: (a) fracture of graphite nodules, (b) yielding and plastic deformation of graphite nodules, and (c) partial tearing away of the matrix from graphite nodules, producing cavities.

Because graphite by itself contributes little to the strength, deformation or fracture of nodules *per se* does not affect the properties. With increased deformation, the graphite/matrix interface area increases. This should result in an increased nucleation rate of ferrite during the subsequent austempering process, reflecting a finer scale of ausferrite. Conversely, the tensile properties in a direction normal to that of deformation would be affected due to the increased area fraction of graphite in the plane of deformation.

In the present work, hot deformation is used in the form of hot rolling on base ductile irons of selected composition and microstructure. The effects of processing variables such as austenitizing temperature and time, austempering temperature and time, deformation temperature, and the amount of deformation on the mechanical properties were studied.

3. Experimental Procedure

3.1 Material Preparation

Table 1 gives the compositions of the different irons used in the study. Preliminary experiments on as-cast materials with ferrite plus pearlite matrix showed that the resulting properties were not uniform. Thus, to have uniform starting material, later experiments were carried out on subcritically annealed material. Subcritical annealing was done by holding the as-cast material at 720 °C for 3 h, followed by furnace cooling to room temperature. After subcritical annealing, the material contained less than 5% pearlite. The material was machined as flat bars of different thicknesses to obtain at least 15-mm-thick bars after the targeted amount of deformation. Because the reductions studied were considerably high, the leading edges were chamfered to a 30 ° angle to facilitate feeding between the rolls. The leading and trailing ends of the bars were discarded, and the material from the middle was used for the tests.

Because the material was rolled straight from the austenitizing furnace, the austenitization temperature itself was considered as the deformation temperature. While lower austenitization temperatures are preferred for better properties in conventional ADI, preliminary rolling trials indicated that even small deformations were not possible below 900 °C. Hence, austenitization temperatures, 900, 950, and 1000 °C, were used in the experiments. Based on the results of previous studies,[12] a uniform austenitizing time of 90 minutes was used, except in the trials where two-step austenitization was used. Rolling was done using a Fenn (United Dominion Co., Newington, CT) rolling mill with H13 rolls of 140-mm diameter and 203-mm width. Austempering temperatures between 260 and 340 °C and austempering times between 30 and 100 min were used in the study. Austempering was completed in a salt bath made up of KNO_3, $NaNO_2$, and $NaNO_3$ in the ratio 55 to 40 to 5.

Table 1 Chemical composition of the irons used

Materials	C	Si	Mn	P	Cu	Ni	Mo	Mg
Material A	3.35	2.62	0.28	0.018	0.97	0.029	0.007	0.048
Material C	3.36	2.42	0.18	0.017	0.22	1.080	0.007	0.040
Material D	3.36	2.43	0.24	0.016	0.60	0.026	0.006	0.050

3.2 Tensile Tests

Tensile test samples were taken from the middle of the austempered bars with axes parallel to the direction of rolling. Samples were machined per ASTM A 370 with 0.252-in. (6.4-mm) gage diameter. Quantitative evaluation of the mechanical properties in directions other than the rolling direction was not completed because of process and material constraints.

4. Results and Discussion

4.1 Tensile Properties

Table 2 shows the different test parameters and the tensile strength, yield strength, and elongation values obtained in the

Table 2 Test parameters and results

Samples	T_γ, °C	t_γ, min	δ_1, %	n_1	$T_{\gamma 2}$, °C	$t_{\gamma 2}$, min	δ_2, %	n_2	T_A °C	t_A min	Tensile strength, MPa	Yield strength, MPa	Elongation %	Quality index, 10^{18} Pa^2
A67	950	90	0	0	...	...	...	...	340	90	1213	923	7.0	10.3
A63	950	90	20	1	...	...	...	...	340	90	1261	875	16.0	25.4
A66	950	90	40	2	...	...	...	...	340	90	1357	923	15.5	28.6
A72	950	90	0	0	...	...	...	...	260	90	1592	1172	2.5	6.3
A71	950	90	20	1	...	...	...	...	260	90	1674	1223	3.3	10.0
A68	950	90	40	2	...	...	...	...	260	90	1791	1275	5.0	16.0
A75	1000	90	50	3	...	...	...	...	260	90	1750	1199	4.5	13.8
A102	950	60	25	1	850	60	...	...	300	30	1297	963	7.0	11.8
C110	950	90	0	0	...	...	...	...	260	90	1268	913	3.0	4.8
C111	950	90	20	1	...	...	...	...	260	90	1499	1009	4.0	9.0
C112	950	90	40	2	...	...	...	...	260	90	1698	944	4.0	11.5
C113	950	90	0	0	...	...	...	...	300	90	1468	1013	4.0	8.6
C114	950	90	20	1	...	...	...	...	300	90	1526	996	5.0	11.7
C115	950	90	40	2	...	...	...	...	300	90	1536	1161	8.0	18.9
C116	950	90	0	0	...	...	...	...	340	90	1199	861	12.0	17.2
C117	950	90	20	1	...	...	...	...	340	90	1189	844	9.0	12.8
C118	950	90	40	2	...	...	...	...	340	90	1247	813	14.0	21.8
D120	900	90	40	2	...	...	...	...	260	90	1702	1302	5.0	15.0
D121	900	90	40	2	...	...	...	...	340	90	1151	868	12.0	15.9
D79	1000	30	40	2	850	75	6	1	275	90	1481	1185	4.0	8.8
D80	1000	30	40	2	850	75	13	1	275	90	1530	1309	2.0	4.7
D84	1000	30	40	2	850	75	10	1	340	90	1089	827	9.0	10.7
D86	1000	30	40	2	900	90	...	...	260	90	1578	1123	5.0	12.5
D88	1000	30	40	2	900	90	...	...	340	90	1130	889	10.0	12.8
D96(a)	950	60	25	1	850	60	...	...	275	30	1171	758	6.0	8.2
D101(a)	950	60	25	1	850	60	...	...	300	30	1213	861	7.0	10.3

T_γ, t_γ: austenitizing temperature and time; $T_{\gamma 2}$, $t_{\gamma 2}$: reaustenitizing temperature and time; T_A, t_A: austempering temperature and time; n_1, δ_1: number of passes and percentage deformation at T_γ; n_2, δ_2: number of passes and percentage secondary deformation at $T_{\gamma 2}$. (a) Without subcritical anneal

tests. Higher strength and lower elongations were obtained with lower austempering temperature (260 °C) compared to those at a higher temperature (340 °C). Properties in the range 1790 MPa/1275 MPa/5% (compared to ASTM A 897M minimum of 1600 MPa/1300 MPa/–) and 1355 MPa/920 MPa/15% (compared to ASTM A 897M minimum of 850 MPa/550 MPa/10) were obtained within the parameter window used in the study. It may be possible to obtain even higher strength values, sacrificing part of the ductility, with the same amount of deformation by austempering below 260 °C. To illustrate the effect of deformation, the variation of tensile strength and elongation with deformation, at the two austempering temperatures for material A, is shown in Fig. 1. Tensile strength and elongation were found to increase with increasing deformation, up to about 40%, beyond which the properties decreased. This is possibly because the graphite nodules at these high deformations got flattened to the extent that the edges were sharp enough to act as stress raisers.

Because in most strengthening treatments the increase in strength normally results in a decrease in ductility and *vice versa,* the two properties should be compared in combination to evaluate the process. The tensile strengths obtained in different tests for three irons used in the study are plotted against elongation along with ASTM standard for ADI in Fig. 2. These figures show that the thermomechanically treated ADI has higher strength than standard ADI, along with higher elongation.

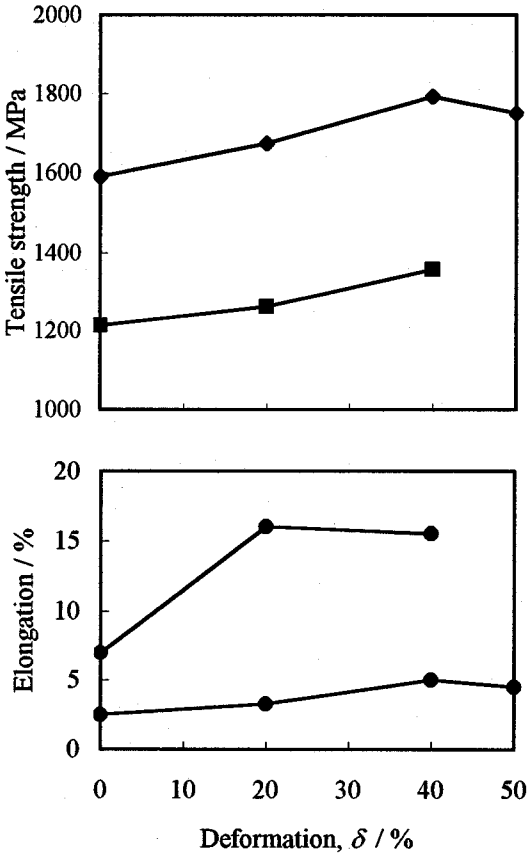

Fig. 1 Variation of tensile strength and elongation of thermomechanically treated irons with deformation

The quality index (QI) of ductile irons takes into account the combined effect of strength and ductility and is defined as[1]

$$QI = (\text{Tensile strength})^2 \bullet (\text{Elongation}, \%) \quad \text{(Eq 2)}$$

The quality indices of the three irons with different test conditions are plotted against elongation in Fig. 3 along with those of standard ADI (ASTM A 897M) for comparison. The identifica-

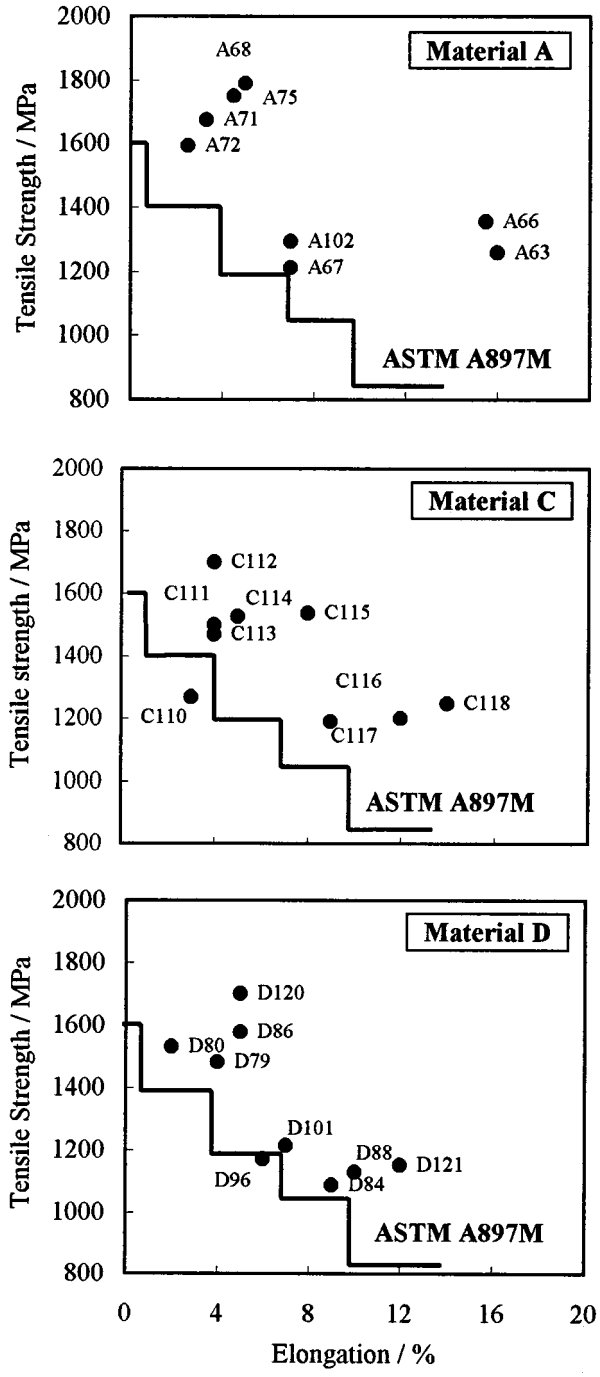

Fig. 2 Tensile strength and elongation values obtained in the experiments compared to ASTM A 897M (refer to Table 2 for test conditions)

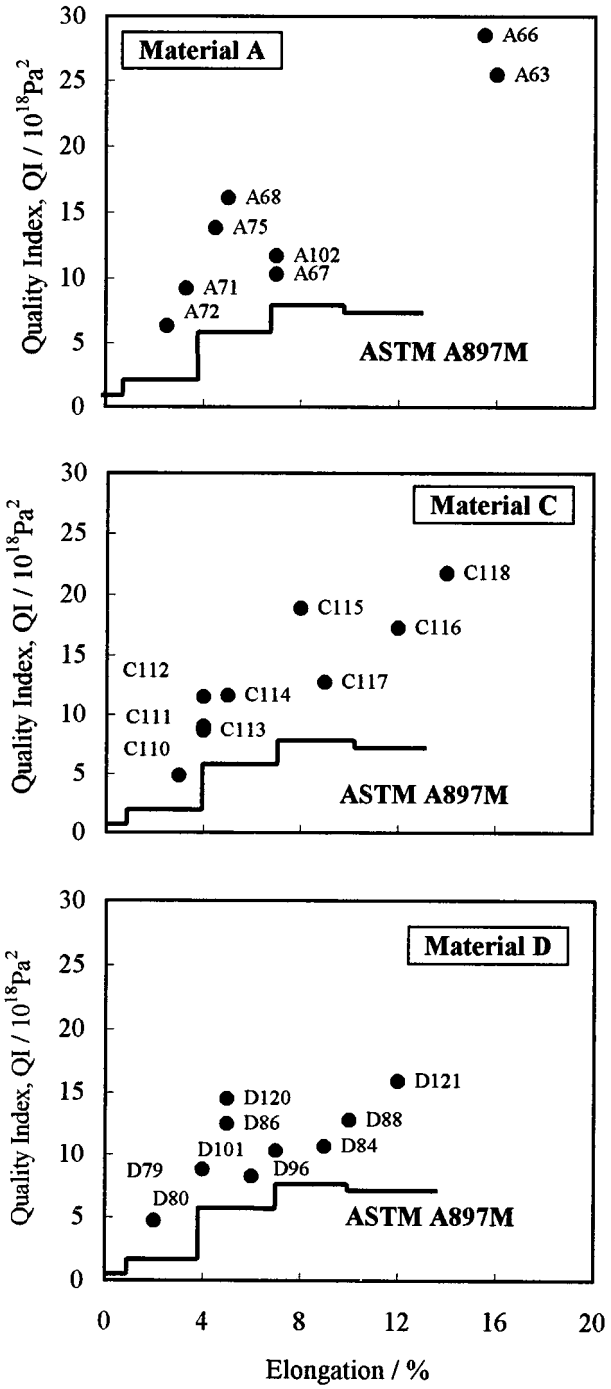

Fig. 3 Quality index values obtained in the experiments compared to ASTM A 897M (refer to Table 2 for test conditions)

tions refer to the sample numbers and corresponding test conditions listed in Table 2. The minimum quality index in standard ADI ranges from 0.5×10^{18} to 7.72×10^{18} Pa^2 with elongations ranging from less than 1 to 10% in the different grades. In comparison, the quality indices obtained in this study range from 4.7×10^{18} to 28.6×10^{18} Pa^2 with elongations between 2.5 and 16%. Higher elongations, and hence higher values of quality indices, were obtained at the higher austempering temperature.

Table 3 Variation of graphite aspect ratio and shape factor with deformation

		Deformation, δ, %					
Parameter	**Plane(a)**	**0**	**10**	**20**	**30**	**40**	**50**
Aspect ratio	LT-ST	0.76	0.66	0.62	0.60	0.52	0.52
	L-ST	0.76	0.63	0.64	0.58	0.54	0.59
	L-LT	0.76	0.74	0.76	0.77	0.75	0.68
Shape factor	LT-ST	0.86	0.82	0.80	0.78	0.74	0.72
	L-ST	0.86	0.79	0.81	0.76	0.75	0.73
	L-LT	0.86	0.83	0.86	0.85	0.84	0.80

(a) LT-ST, long transverse-short transverse; L-ST, longitudinal-short transverse; L-LT, longitudinal -long transverse.[13]

4.2 Reaustenitization and Secondary Deformation

Because ADI has higher hardness compared to as-cast and annealed irons, it may be worthwhile to explore the possibility of completing the machining operations before the austempering treatment, but after the grain refining deformation. For this, the material should be normalized after the deformation, machined, reaustenitized, and subjected to austempering treatment. The reaustenitization temperature can be lower because no deformation needs to be done at this temperature, and the matrix austenite carbon content will be lower at lower temperatures. Additional refinement can be achieved by small amounts of final deformation at austempering temperature during the early minutes of austempering (in the incubation period). The structure after such secondary deformation was found to be very fine, and at 13% deformation at austempering temperature (in addition to 40% at austenitizing temperature), the material had good tensile properties (1530 MPa/1310 MPa/2%). The microstructure in all reaustenitized samples contained a substantial amount of ferrite. This may be due to localized regions not becoming fully austenitized due to lower temperature.

4.3 Graphite Deformation

Change in graphite shape became visually noticeable only beyond 15% deformation. At deformations beyond about 40%, the nodule edges became sharp enough to act as stress raisers, explaining some decrease in strength at 50% deformation and surface cracking at 60% deformation. Once deformed, the shape of the graphite remained the same during the austempering treatment. The nodules were flattened in the direction of roll pressure and elongated in the rolling direction and hence had the highest area fraction in the rolling plane. Maximum change in shape was observed in the other two perpendicular planes. To obtain a quantitative evaluation of graphite deformation, image analysis was done on samples with different amounts of deformation, and the aspect ratio and shape factor were determined in three mutually perpendicular planes. Table 3 gives the results, which show that minimum changes in aspect ratio and shape factor occurred in the rolling plane. It was seen in the microstructures that along this plane, the graphite nodule sections were almost circular even at 50% deformation, but with much larger area fraction than the other two perpendicular planes.

5. Conclusions

The following conclusions can be drawn.

- Subcritical annealing is necessary to obtain consistent results after the thermomechanical treatment.
- The properties obtained in thermomechanically treated ductile iron are highly sensitive to the general quality of the as-cast material.
- A higher austempering temperature offers higher ductility and lower strength values due to the combined effect of higher volume fraction and higher carbon content in saturated austenite. Conversely, lower austempering temperature offers higher strength at lower ductility due to higher volume fraction and more refined ausferrite.
- Up to 40% deformation, strength and elongation progressively increased with deformation. Beyond 40% deformation, both properties started decreasing due to graphite nodules getting flattened to the extent that their edges acted as regions of stress concentration.
- The rate of increase in strength and ductility and hence in QI with deformation is higher at lower austempering temperatures.
- Lower austenitization temperatures offer higher strength and elongation, but below 900°C, the properties start decreasing due to the presence of residual ferrite.
- While the properties were still better than those without deformation, reaustenitization at a lower temperature after deformation at a higher temperature did not produce the same improvements in strength and ductility due to the presence of residual ferrite.

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