EFFECT OF AUSTEMPERING PARAMETERS ON MICROSTRUCTURE OF ADI WITH 1.5% Ni

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Abstract

Ductile iron with 1.56% Ni was subjected to various cycles of the austempering treatment. For all variants, the austenitization temperature was 920oC and the time of holding was 2 hours. Austempering was carried out at temperatures of 260, 300, 330, 360, 380, 400oC with the time of 15, 30, 60, 90, 120 and 150 minutes. After each variant, the microstructure of the metal matrix was examined. Measurements covered hardness of the matrix and microhardness of "acicular" ferrite, austenite and martensite

Keywords: Ductile iron, heat treatment, the microstructure of ADI, ADI properties

1. INTRODUCTION

ADI (Austempered Ductile Iron) is spheroidal graphite cast iron subjected to austempering treatment. The process of obtaining ADI consists of several steps, which are schematically shown in **Figure 1**. The aim of the process is to obtain the microstructure of cast iron matrix that is called ausferrite and consists of a mixture of ferrite in the form of plates, similar to "needles" occurring in bainite, and thermodynamically stable austenite with high carbon content [1, 2].

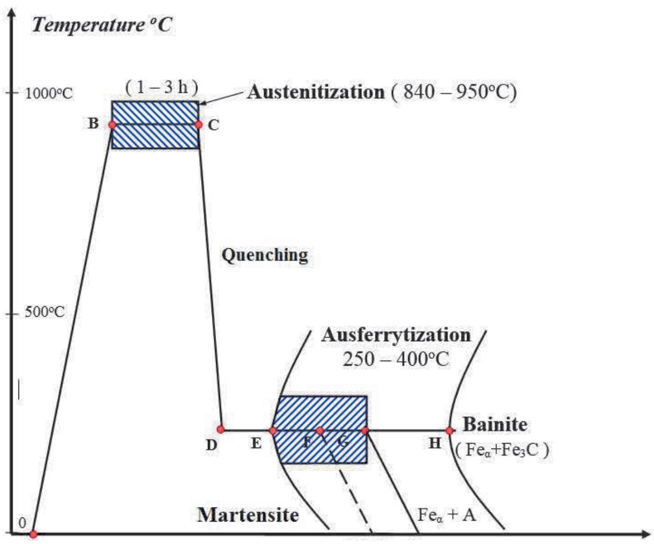


Figure 1 Schematic diagram of the austempering heat treatment cycle [1]

In ADI technology, the casting is subjected to austenitizing treatment by heating it to a temperature in the range of 840-950°C and holding at this temperature until the whole matrix is converted into carbon-saturated austenite (BC line in **Figure 1**). Then the casting is cooled at a high speed (C-D) to the temperature of isothermal transformation (230-400°C). At point E, the precipitation of ferrite starts taking place. During holding at the temperature of isothermal transformation, further transformation of austenite takes place. At point F, the "acicular" ferrite starts precipitating. In the range between points E and F, carbon diffuses to the remaining austenite, increasing its content from 1.2 to 1.6%. According to Kovac [1], such carbon content makes the austenite metastable, which means that it can exist in the matrix at room temperature, but not necessarily will be stable. If the casting is cooled down to a point below room temperature, or if mechanical stresses are formed as a result of, e.g., machining, this type of transformation will cause problems during machining, dimensional changes and loss of ductility. In contrast, ferrite nucleation in the F-G range is of no major importance. Ferrite grains continue growing, and this effect is accompanied by simultaneous diffusion of carbon to residual austenite, raising carbon concentration in the austenite from 1.8 to 2.2%. At this level of carbon concentration, the austenite becomes stable both thermally and mechanically [1,2].

This paper presents the results of research on the effect of heat treatment parameters on microstructure of the ADI with 1.56% Ni. The results presented complement and extend the research described in [3]. Matrix components were classified, their volume fractions were measured along with the measurement of matrix hardness and microhardness, and optimum heat treatment parameters were determined to obtain ADI with the chemical composition as given in **Table 1**.

2. TEST MATERIAL

Spheroidal graphite cast iron of the chemical composition given in **Table 1** was used for the tests and was subjected to various cycles of the austempering treatment. Fixed austenitizing conditions were chosen (temperature: $T_\gamma = 920$ °C, time: $t_\gamma = 120$ min.) and, basing on the TTT diagram, 18 variants of the austempering treatment were established [3]. When selecting the temperatures of the austempering treatment, the bainitic transformation area was divided into 2 ranges: upper and lower (**Table 2**).

Table 1 Chemical composition of the tested spheroidal graphite cast iron.

C [%]	**Si** [%]	**Mn** [%]	**Mg** [%]	**Ni** [%]	**P** [%]	**S** [%]
3.55	2.55	0.31	0.063	1.56	0.025	0.009

Table 2 Variants of the austempering treatment

	Variant	T [°C]	t [min.]	**Variant**	T [°C]	t [min.]	**Variant**	T [°C]	t [min.]
upper range	**W11**	400	15	**P11**	400	60	**W12**	400	90
	W9	380	15	**P9**	380	60	**W10**	380	90
	W7	360	15	**P7**	360	60	**W8**	360	90
lower range	**Variant**	T [°C]	t [min.]	**Variant**	T [°C]	t [min.]	**Variant**	T [°C]	t [min.]
	W5	330	30	**W6**	330	120	**P5**	330	150
	W3	300	30	**W4**	300	120	**P3**	300	150
	W1	260	30	**W2**	260	120	**P1**	260	150

3. TEST METHODS

Metallographic studies - metallographic sections of ADI specimens were etched with NITAL reagent (1% HNO3 nitric acid solution in C2H5OH ethyl alcohol) and were next examined under a ZEISS Axio Imager M1m light microscope.

Computer image analysis - image analysis was performed with a Metilo program. The volume fraction of each matrix phase was determined as an average of 4 measurements taken for each variant of the heat treatment. The preparation of the image for automatic measurement included the use of a medium grey filter and an automatic K-median binarization. In some cases, it was necessary to correct the shadow and manually set the grey threshold.

Hardness measurement - Brinell hardness measurements were taken with an INNOVATEST / Nexus 703 A hardness tester. The test consisted in pressing a calibrated ball of D = 2.5 mm diameter under a pressure of

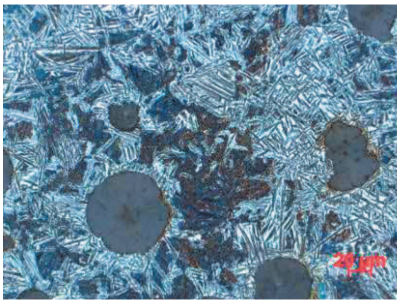
P = 187.5 kG against a polished surface of the sample. The full time of pressing was 15 s. For each variant, 15 measurements were taken by Brinell method, and then average values were calculated.

Microhardness measurement - microhardness tests were performed using a SHIMADZU HMV-G microhardness gauge. The indenter was a pyramid with an angle of 136 degrees. For each sample, five indentations were made in the matrix using a 100g load (HV0,1).

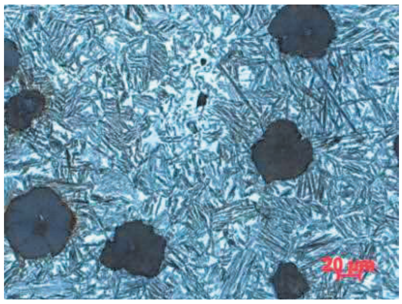
Nanohardness measurement - the nanohardness of the individual microstructural constituents was measured with a CSM Instruments nanohardness tester. A Berkovich type diamond indenter and a pressure of 50 mN were used. Three measurements were taken for each microstructural constituent in a given variant, calculating next an average.

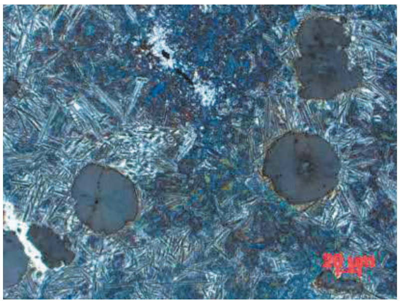
4. TEST RESULTS

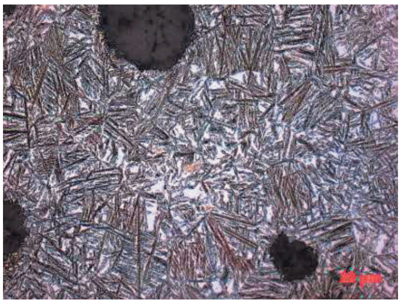
Figures 2 and **3** show photographs of microstructures of the tested cast iron at 500x magnification. **Figure 2** shows microstructures obtained after austempering in the upper range of temperatures, and **Figure 3** after austempering in the lower range of temperatures.

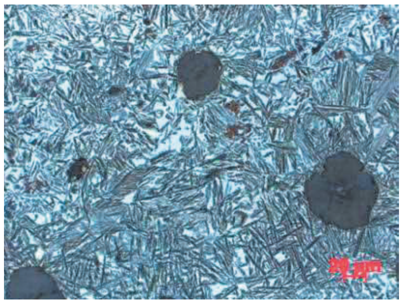

a) W11 (T = 400 °C, t = 15 min.)

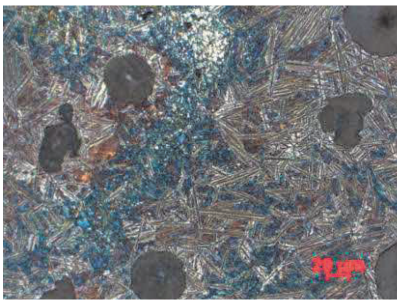

b) P11 (T = 400 °C, t = 60 min.)

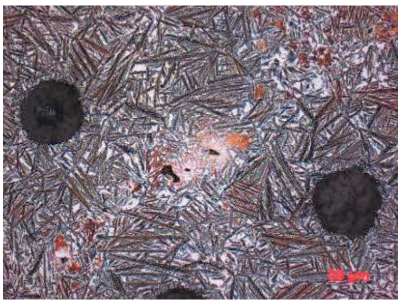

c) W12 (T = 400 °C, t = 90 min.)


d) W9 (T = 380 °C, t = 15 min.)


e) P9 (T = 380 °C, t = 60 min.)


f) W10 (T = 380 °C, t = 90 min.)


g) W7 (T = 360 °C, t = 15 min.)


h) P7 (T = 360 °C, t = 60 min.)

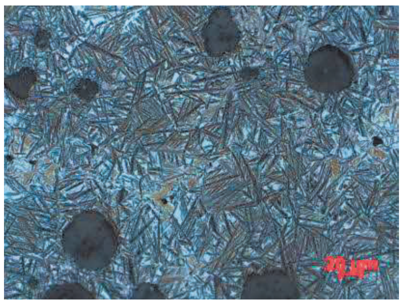
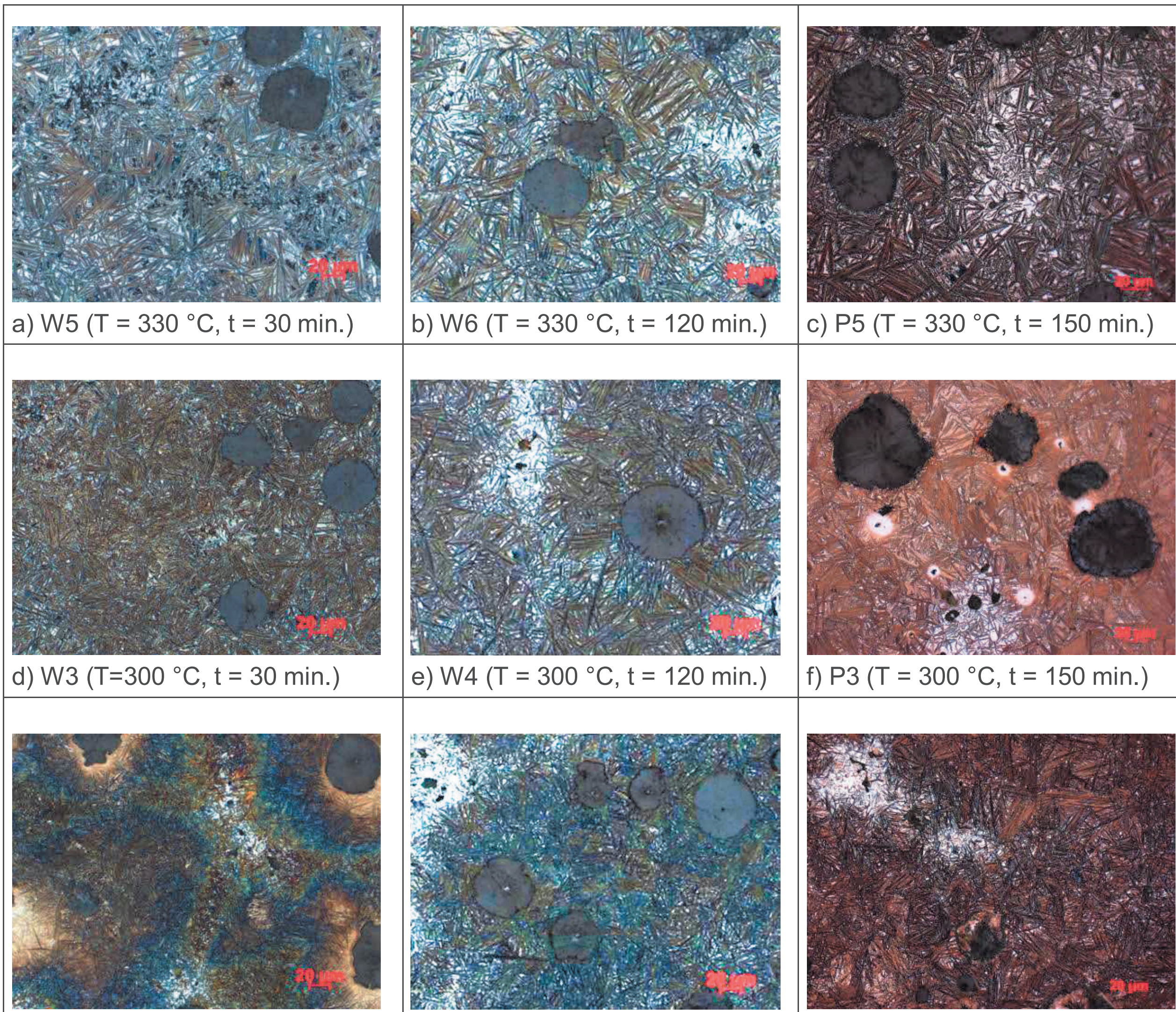

i) W8 (T = 360 °C, t = 90 min.)

Figure 2 Microstructure of the tested cast iron - upper range (500x)



a) W5 (T = 330 °C, t = 30 min.) b) W6 (T = 330 °C, t = 120 min.) c) P5 (T = 330 °C, t = 150 min.)
d) W3 (T=300 °C, t = 30 min.) e) W4 (T = 300 °C, t = 120 min.) f) P3 (T = 300 °C, t = 150 min.)
g) W1 (T = 260 °C, t = 30 min.) h) W2 (T = 260 °C, t = 120 min.) i) P1 (T = 260 °C, t = 150 min.)

Figure 3 Microstructure of the tested cast iron - lower range (500x)

The results of measurements of the volume fractions of austenite are presented for the upper and lower range in **Tables 3**, while the results of hardness and microstructure measurements are presented for the upper and lower range in **Tables 4**.

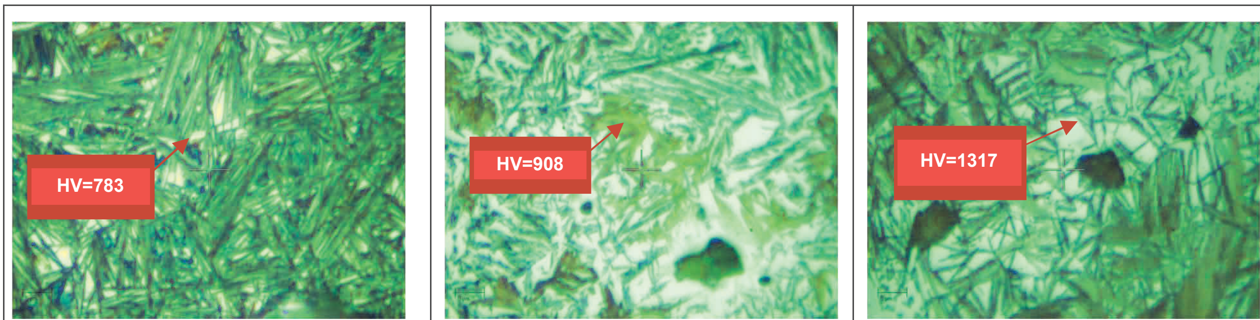
Table 3 Volume fraction of austenite

	Variant	Vva [%]	Variant	Vva [%]	Variant	Vva [%]
upper range	**W11**	10.9	**P11**	23.4	**W12**	20.5
	W9	5.2	**P9**	17.3	**W10**	16.6
	W7	3.5	**P7**	13	**W8**	13.9
lower range	**Variant**	Vva [%]	**Variant**	Vva [%]	**Variant**	Vva [%]
	W5	6.2	**W6**	13.1	**P5**	7.3
	W3	5.1	**W4**	8.6	**P3**	4
	W1	1.67	**W2**	7.6	**P1**	3

Table 4 Hardness and microhardness

	Variant	HBW	HV0,1	Variant	HBW	HV0,1	Variant	HBW	HV0,1
upper range	**W11**	292	449.4	**P11**	236.1	370.6	**W12**	270.13	425.0
	W9	368.73	430.2	**P9**	251	393.6	**W10**	284.33	410.8
	W7	400.6	431.4	**P7**	168.22	413.4	**W8**	324.87	378.2
lower range	**Variant**	**HBW**	**HV0,1**	**Variant**	**HBW**	**HV0,1**	**Variant**	**HBW**	**HV0,1**
	W5	375	481.8	**W6**	370.8	460.0	**P5**	282.11	488.6
	W3	436.5	589.6	**W4**	391.73	503.0	**P3**	329.58	478.0
	W1	527.27	614.0	**W2**	451.47	536.6	**P1**	353.79	495.4

Nanohardness of selected variants are presented at **Figure 4**.



a) W3 (T = 300 °C, t = 30 min.) Results of measurements: 703, 783, 839

b) P7 (T = 360 °C, t = 60 min.) Results of measurements: 908, 922, 939

c) P3 (T = 300 °C, t = 150 min.) Results of measurements: 2405, 1317, 1607

Figure 4 Nanohardness of selected microstructural components

5. DISCUSSION OF RESULTS

Chemical composition of base cast iron used for the manufacture of ADI is typical of pearlitic-ferritic spheroidal graphite cast iron. To raise the cast iron hardenability, only one element was added i.e. about 1.5% nickel (**Table 1**). This is not a typical composition, as usually in these types of cast iron, to improve the hardenability, combinations of several elements, such as Ni, Mo, Cu, sometimes Mn and Cr, are applied. In the technical literature there is no data on this type of cast iron. However, Benam [4] suggest that studies of ADI with the addition of Ni might be interesting.

Different variants of the austempering treatment were applied (**Table 2**). The time of 15 and 30 minutes is unlikely to be used in practice, but here it has been used for cognitive reasons.

Hardness measured by the Brinell method and HV microhardness show large scatter of results depending on the heat treatment variant used (**Table 4**). The highest HBW values were obtained in variants W1 (527.3) and W2 (451.5). Hardness decreases with an increase of the austempering temperature. Similar effect is observed for the austenite volume fraction (**Table 3**). Large amounts of austenite (23.4% for variant P11 and, e.g., 13.1 for variant W6) demonstrate high ductility of ADI. Nanohardness measurements carried out for selected heat treatment variants showed extreme hardness values from 1600 to 2400. Such values were obtained in the white fields shown in, e.g., **Figure 4**. This indicates that only martensite with very fine "needles" could occur in those places.

As referred to Kovac research [1], the analysis of images combined with microhardness measurements indicates that the matrix microstructure characteristic of ADI was obtained in variants W12 and W10. The classification of ADI is based on the strength properties (standard PN-EN 1564, ASTM A897), so it seems necessary to carry out endurance tests for these variants to determine the grades obtained. For the short-time austempering of 15 min (upper range) and 30 min (lower range), the matrix microstructure typical of ADI has not been obtained. Studies suggest that as a result of the austempering time too short, austenite has not been evenly saturated with carbon, and as a consequence of rapid cooling, the transformation into martensite has occurred. As suggested by Kovac [1], martensite of this type, formed by the achievement of local temperature Ms during temperature cycle, undergoes tempering and its hardness is inferior to the hardness of the martensite formed as a result of, e.g., the stresses mechanically-induced by machining (**Figure 4a**). In variants P11, P9, P7 and W8, the matrix microstructure consists mainly of ausferrite, though traces of martensite also appear there (**Figure 4b**). In photographs taken for the cast iron austempered according to variants W6, W4, W2, P5, P3, P1 are clearly visible the fields of unreacted metastable austenite, which during austempering did not participate in the reaction but its carbon content has not changed in the treatment cycle leaving it metastable [1]. As a result of mechanical processing, due to the stresses formed, this austenite can transform into martensite. Martensite formed from the metastable austenite is characterized by very high hardness, as confirmed by the nanohardness tests (**Figure 4c**). (As a result of pressure exerted by the indenter, the austenite could be transformed into martensite).

Analysis of the hardness measurements of the matrix microstructure confirms the research conducted. For the upper range and the time of 15 min (W11, W9, W7), the hardness value is high, which can be due to the presence of martensite in the microstructure. For the time of 60 min, hardness assumes the lowest value, which may result from the austenite saturation with carbon lower than in the case of longer times. In the lower range, on the other hand, a decrease in hardness is observed with the increasing time of isothermal holding. The nature of this phenomenon is confirmed by the results of microstructure analysis. For variants W5, W3 and W1, the matrix microstructure is in prevailing part composed of martensite. For variants W6, W4 and W2, the martensite content is already much lower.

6. CONCLUSION

ADI with the addition of only one alloying element is cheaper than its counterpart with the addition of two or three elements. Test results have proved that it is possible to obtain ausferritic matrix microstructure in the starting spheroidal graphite cast iron containing only Ni as an alloying addition. Based on the results of the studies, only 2 variants that meet this condition have been selected. In order to classify the tested cast iron, tests of the tensile strength Rm and elongation A5 should be carried out.

The obtained results will be used to verify the mathematical model for the analysis of the development of spheroidal iron microstructure during heat treatment. The results of the study can also be used as a source of knowledge in computer systems that are developed for predicting the structure and properties of alloys after casting processes. Especially that the computer technologies, being currently available as a cheap and effective way of optimization, are used for modelling and analysis of phenomena occurring in many areas of research [5, 6].

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REFERENCES

[1] KOVAC, B. V. On the terminology and structure of ADI. *AFS Transactions*, 1994, vol. 102, pp. 417 - 420.

[2] GUZIK, E. SOKOLNICKI M., NOWAK A., Effect of heat treatment parameters on the toughness of unalloyed ausferritic ductile iron. *Archives of Foundry Engineering*, 2016, vol. 16, no. 2, pp. 79-84.

[3] MRZYGŁÓD, B., OLEJARCZYK-WOŻEŃSKA, I., MATUSIEWICZ, P., KOPYŚCIAŃSKI, M., GŁOWACKI, M. Quantitative analysis of selected parameters of the microstructure of ADI with the addition of Ni. In *METAL 2016: 25th International Conference on Metallurgy and Materials.* Ostrava: TANGER, 2016, pp. 795-800.

[4] BENAM, A. S. Effect of alloying elements on austempered ductile iron (ADI) properties and its process: Review, *China Foundry*, 2015, vol. 12, no. 1, pp. 54 - 70.

[5] KLUSKA-NAWARECKA, S., REGULSKI, K., KRZYŻAK, M., LEŚNIAK, G., GURDA, M. System of semantic integration of non-structuralized documents in natural language in the domain of metallurgy, *Archives of Metallurgy and Materials*, 2013, vol. 58, no. 3, pp. 927-930.

[6] HOJNY, M., GŁOWACKI, M. Modeling of Strain-Stress Relationship for Carbon Steel Deformed at Temperature Exceeding Hot Rolling Range, *J. Eng. Mater. Technol*, 2011, vol. 133, no. 2 pp. 021008-1-021008-7.