Examination of Cast Iron Material Properties by Means of the Nanoindentation Method

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Abstract

The paper presents results of examination of material parameters of cast iron with structure obtained under rapid resolidification conditions carried out by means of the nanoindentation method.

Keywords: Cast iron, Rapid crystallization, Indentation test

1. Introduction

The method of improving service properties of cast iron through superficial remelting with the use of Gas Tungsten Arc Welding (GTAW) [1–7] gains increasing popularity. To determine results obtained by means of this process, hardness measurements are typically used. More knowledge of the material parameters can be provided by indentation tests that allow to obtain information about the modulus of elasticity, mechanical work, relaxation, or creeping of the material. The standard hardness testing procedures are based on material hardness observed after the load being removed from the indenter. The instrumented indentation method used here allows to assess the course of force change and displacement of the indenter while penetrating the examined material in the course of both loading and unloading [8–20]. Indentation tests are carried out with the use of Vickers or Berkovich diamond cones under given indentation force and speed, followed by a pause of definite duration and unloading of the indentation force with given speed. Elastic displacement of the indenter is governed by the Sneddon’s equation [15]. Theoretical foundations of indentation tests were given by Olivier and Pharr [10]. The test result is obtained in the form of a plot representing the load force versus displacement functional dependence. Based on the plot, one can calculate: hardness, modulus of elasticity and mechanical work relating to forcing indenter into the material (elastic deformation work and plastic deformation work). In Figure 1, an example indentation test curve is shown.

![Indentation Test Curve](image)

Fig. 1. Schematic recess indenter (a) and schematic curve recorded during the indentation test (b) [7-9,15]:

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1 -the line of sample surface permanent deformation caused by the indenter after load release, 2 -the line of sample surface permanent deformation at maximum indentation \( h_{\text{max}} \) with maximum force \( F_{\text{max}} \), A -application of the load force \( F \), B -removal of the load force \( F \), \( h_c \) -the depth of contact of the indenter with the sample at \( F_{\text{max}} \), \( h_p \) -the depth of the indenter penetration after release of \( F_{\text{max}} \), \( h_r \) -point of intersection with the tangent to the unloading curve \( B \), \( W_{\text{spr}} \) -the elastic deformation work, \( W_{\text{plast}} \) -the plastic deformation work

2. Methods

2.1. Material for study

The research work was carried out with unalloyed spheroidal cast iron in the condition after surface remelting by means of GTAW method. The chemical composition analysis was carried out with the use of Q4-TASMAN emission spectrometer by Bruker. The cast iron contained 3.49% C; 2.30% Si; 0.66% Mn; 0.019% S; 0.039% P; 0.17% Cu; 0.01% Ni; 0.084% Mg. The tests were carried out on castings in the form of plates with dimensions 200×50×10 mm. On the castings, superficial remeltings were made with the use of plasma of electric arc generated by means of TETRIX 351 AC/DC welder by EWM. The remeltings were made in argon atmosphere at welding current intensities \( I = 50, 100, 200 \) and 300 A with the electric arc scanning rate \( v_s = 200 \) mm/min. The test samples were ground and polished. Designation of test samples and parameters of the remelting process are presented in Table 1.

Table 1.
Determination of test samples and the parameters of the GTAW

| No | Welding current, \( I \) [A] | The electric arc scanning rate, \( v_s \) [mm/min] |
|----|-----------------|------------------|
| A  | 300             | 200              |
| B  | 300             | 400              |
| C  | 300             | 600              |
| D  | 300             | 800              |

2.2. Indentation test

The cast iron indentation tests were carried out on a test stand equipped with a multi-function platform OPX NHT/NST by CSM Instruments with a Berkovich indenter with a tip diameter 2 μm and angle 90°. The measurement head allows to carry out tests in the load force range from 0.1 mN to 500 mN. The test set-up is presented in Figure 2.

Cast iron samples were examined by means of nanoindentation method with load force \( F_N = 450 \) mN and the load followed by a 15 s pause. The load application and removal rates both equalled to 900 mN/min. The indentation tests were carried out as per ASTM [8] and PN EN ISO [9] standards. In Figure 3, plots of force \( F_N \) value is presented versus indenter displacement \( P_d \) in the cast iron obtained under rapid resolidification conditions.

In Table 2, measurement results obtained from the indentation test for cast iron obtained under rapid resolidification conditions. Figure 4 presents the indentation test results in graphical form.

3. Analysis of results

Cast iron obtained under rapid resolidification conditions is characterised with diversified microstructure [21–24]. The effect of increased of the welding current intensity consists in larger volume of the molten metal pool which results in lower solidification rate. This in turn is related to a lower degree of fragmentation of the cementite eutectic in which, as the result of rapid cooling down to ambient temperature, austenite is partly transformed into hardening product.
Table 2.
Indentation test results obtained on the structure of iron under conditions of rapid crystallization

| No | Melting parameters | Results of indentation |
|----|-------------------|------------------------|
|    | welding currents  | speed $v_s$ [mm/min] | $h_{\text{max}}$ [nm] | $h_c$ [nm] | $h_r$ [nm] | $h_p$ [nm] | $W_{\text{spr}}$ [pJ] | $W_{\text{plast}}$ [pJ] | $W_{\text{total}}$ [pJ] | $\eta_{\text{IT}}$ [%] | $E_{\text{IT}}$ [N/mm$^2$] | HV |
| A  | 300              | 200                    | 1676                  | 1607      | 1467      | 76407         | 76407                 | 192835                | 269242               | 28,38                 | 185                    | 649 |
| B  | 300              | 400                    | 1279                  | 1196      | 1070      | 93294         | 93294                 | 167077                | 260371               | 35,83                 | 191                    | 1114 |
| C  | 300              | 600                    | 1207                  | 1129      | 1005      | 87300         | 87300                 | 143453                | 230753               | 37,83                 | 223                    | 1249 |
| D  | 300              | 800                    | 1180                  | 1099      | 969       | 88934         | 88934                 | 144264                | 233198               | 38,14                 | 224                    | 1307 |

where: $h_c$ - depth of indenter-sample contact at $F_{\text{max}}$; $h_r$ - point of intersection with the tangent to load removal curve $B$ (Fig. 1); $W_{\text{spr}}$ - elastic deformation work; $W_{\text{plast}}$ - plastic deformation work; $W_{\text{total}} = W_{\text{spr}} + W_{\text{plast}}$; $\eta_{\text{IT}} = (W_{\text{spr}}/W_{\text{total}}) \times 100$ - elastic force work share in the loading/unloading cycle

Fig. 4. Effect of HV hardness and elastic module $E_{\text{IT}}$ cast iron structure resulting in a rapid crystallization of the indenter penetration of a, b), mechanical work c, d), the share of the work of elastic e, f)

The molten metal pool volume has also an effect on the rate at which cementite eutectic cools down to ambient temperature and thus on the volume share of hardening products and the volume share of residual austenite. This manifests itself in the material hardness diversification from 649 HV in the case of remelting performed with the electric arc scanning rate of $v_s = 200$ mm/min to 1307 HV in the case of remelting carried out by the scanning with electric arc at rate $v_s = 800$ mm/min.

Cast iron with structure obtained under rapid resolidification conditions resulting from surface remelting with the use of higher electric arc scanning rates is characterised with higher values of the modulus of elasticity $E_{\text{IT}}$. They equal $E_{\text{IT}} = 185$ N/mm$^2$ at the electric arc scanning rate of $v_s = 200$ mm/min and
E_{IT} = 224 \text{ N/mm}^2 \text{ at the electric arc scanning rate of } v = 800 \text{ mm/min, respectively. Values of material parameters are reflected in course of the curve representing penetration of indenter into the material during applying and removing the load (geometrical parameters of the indentation process, } h_{\text{max}}, h_c, h_r). \text{ Analysis of course of the indentation curve shows that for remeltings obtained with the use of higher electric arc scanning rates, the total work value decreases. This is a result of a decrease of value of work relating to the plastic force. With increasing velocity of electric arc scanning, the molten metal pool volume decreases which results in increased pace of resolidification. This in turn results in decrease of the inter-phase distance } \lambda \text{ in cementite eutectic and increase of share of the hardening products and thus a lower share of austenite in the material structure [21–24]. The changes occurring in the microstructure are reflected in the increase of share of the elastic force work } W_{\text{spe}} \text{ in the total work } W_{\text{total}} \text{ from } \eta_{IT} = 28.38\% \text{ for the electric arc scanning rate } v = 200 \text{ mm/min to } \eta_{IT} = 38.14\% \text{ for the for the electric arc scanning rate } v = 800 \text{ mm/min.}

4. Conclusions

The obtained results indicate that the tests carried out with the use of the nanoindentation method is an effective tool useful in evaluation of material structure parameters that can be further used for assessment of service properties of materials.

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