Non-destructive evaluation of the micro residual stresses of IIIrd order by using micro magnetic methods

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Abstract

Nanoscale coherent precipitates and the corresponding micro residual stresses play a dominant role in the strengthening process of materials. At present, there exists no experimental method for measuring micro residual stresses of IIIrd kind non-destructively. In the frame of the present work, it will be shown that micro-magnetic measurement techniques based on the tensile loading dependent maximum Barkhausen noise amplitude can be used for the analysis of micro residual stresses (MRS) of IIIrd kind (coherency residual stresses). For this purpose, Fe-Cu-alloys with well-defined contents of Cu-precipitates were produced and investigated.

1. Introduction

Residual stresses are self-equilibrating stresses existing in materials under uniform temperature conditions and without external loading. The inner mechanical forces and moments combined with the micro residual stresses are balanced in a mechanical equilibrium [1]. Measurement of MRS in materials is of increasing interest. Generally speaking, MRS in superimposition with load induced stresses have advantages when they compensate. MRS change under the influence of the temperature/time conditions. Due to their capability to detect the change in the interaction between the magnetic structure and the lattice defects like dislocation, precipitates, grain boundaries or residual stress fields, the micro-magnetic ND techniques are suitable for the characterization of the residual stress state of the component.

One of the objectives of the present research work was to develop a micro-magnetic non-destructive method as an alternative to the time consuming radiographic method for quantitative characterization of the MRS of IIIrd order in iron-based materials.

2. Theoretical Background

2.1 *Magnetic methods - Barkhausen noise measurements*

Ferromagnetic materials consist of small, finite regions called magnetic domains. Each domain is spontaneously magnetized to the saturation value M_S (saturation magnetisation) of the material. These domains are separated by Bloch walls [2]. Two kinds of Bloch walls are observed in the magnetic structure of a ferrous magnetic material: 180°-Bloch walls and 90°-Bloch walls. The indicated angle is the angle between the magnetization vectors in two adjacent domains. During magnetization the Bloch wall movement takes place discontinuously because the Bloch walls are temporarily pinned by microstructural obstacles like dislocations, precipitates, phases or grains boundaries in a polycrystalline material. The stepwise breakaway of the Bloch walls from the obstacles changes the magnetization state locally. The local magnetization changes induce pulsed eddy currents in the vicinity of the events. The pulsed eddy currents induce electrical voltage pulses, detected by an induction coil surrounding the magnetized specimen, the so called Barkhausen noise [2]. After suitable rectification, amplification and filtering the noise signals are plotted as function of the tangential field strength H_t. A typical Barkhausen noise profile $M(H_t)$ is shown in Figure 1.

Fig.1: Hysteresis curve and magnetic Barkhausen noise profile $M(H_t)$-curve

By measuring the maximum amplitudes of the Barkhausen noise signals M_{MAX} the stress states of the ferrite phase of steel can be analyzed. MRS of IIIrd order interact with the magnetic structure. The micro-magnetic theory appoints that the macroscopic hysteresis is produced by the microscopic Bloch wall movements and their interaction with the microstructure and stress fields.

2.2 *Integral Load-Stress Related Barkhausen Noise*

The micro-magnetic concept to characterize MRS is based on load-stress-dependent Barkhausen noise measurements [3], [4]. The maximum of the Barkhausen noise amplitude obtained during one hysteresis cycle is recorded as a function of the load induced tensile load stress. These series of

Barkhausen noise maxima related to the applied tensile stress again reach a relative maximum. A shift of this relative maximum along the stress axis can be observed as a measure for the change of the micro (or macro) residual stress state. A measurement technique based on this effect permits the quantitative characterization of residual stress without the use of a reference method such as X-Ray diffraction [3]. So far the superimposed residual stress is of the tensile type, the Barkhausen noise activity of the iron-based materials is more enhanced than in the stress-free state and the curves reach their maximum sooner, i.e. the curve shifts to the left-hand side (Fig. 2, grey curve) and in the other direction in case of the superimposed compressive stresses (Fig. 2, dotted grey curve).

Fig. 2: Schematically represented shift of the tensile load stress dependence of the maximum Barkhausen noise amplitude ($M_{MAX}(\sigma)$-curve) for a higher tensile residual stress state (grey curve) and for a higher compressive residual stress state (dotted grey curve)

In case of a material which contains compressive MRS superimposed with tensile MRS the maximum of the $M_{MAX}(\sigma)$ curve shifts as follows:

1) The occurrence of the compressive MRS cause a shift of the maximum of the $M_{MAX}(\sigma)$ curve in comparison to the stress-free state (black curve) to the right-hand side along the load stress axis.
2) Further on the occurrence of the tensile MRS causes a shift of the maximum of the $M_{MAX}(\sigma)$ curve in comparison to the stress-free state to the left-hand side along the load stress axis.

3. Investigated materials

For the present study binary Fe- Cu alloys were investigated. In order to obtain precipitation hardened alloys, a typical heat treatment was performed: (1) solution annealing in the monophase area at 850°C for 2 hours in a vacuum furnace, (2) quenching into water and (3) thermal ageing in the two-phase area at 500°C for 390 minutes and 1500 minutes [5-9]. The temperature and time parameters were determined by means of Monte-Carlo simulation at Institute for Materials Testing, Materials Science and Strength of Materials, University of Stuttgart [10].

During the thermal ageing Cu precipitates nucleate, grow and change their microstructure from b.c.c. (coherent with the Fe matrix) into f.c.c. (incoherent with the Fe matrix). Small and coherent precipitates cause the increase of the hardness and of the MRS of IIIrd order whereas incoherent precipitates cause the decrease of the hardness and of the MRS of IIIrd order (Fig. 3).

Figure 3: Comparison between the hardness (HV5) and the micro residual stresses during increasing thermal ageing time for the Fe-1.0wt.%Cu

Small angle neutron scattering (SANS) measurements performed after thermal ageing for 390 minutes are presented in the table 1 and show that the thermal ageing corresponding to the maximum of the hardness caused coherent Cu particles. Cu particles having a radius smaller the 4 nm are coherent with the surrounding Fe matrix.

Table 1: Results from the SANS measurements on Fe-1.0wt.%Cu after thermal ageing at 500 °C / 390 min

The MRS of IIIrd order are caused by the mismatch in the lattice parameters between Cu and Fe. The fact that the lattice parameter of Cu is larger than that of Fe, causes coherent micro residual tensile stresses of IIIrd order in the α-Fe matrix. The MRS of IIIrd order increase during nucleation and formation of the coherent Cu particles and decrease during the transformation of the coherent Cu particles into incoherent Cu particles.

Additionally to the MRS of IIIrd order the precipitation of Cu causes thermal induced MRS of IInd order. The MRS of IInd order are caused by the difference between the thermal expansion coefficient of Fe and Cu. The fact that the thermal expansion coefficient of Cu is larger than that of Fe, causes compressive MRS of IInd order in the α-Fe matrix. The MRS of IInd order don't change during the thermal ageing.

4. Experimental setup

In order to evaluate MRS load-dependent Barkhausen noise measurements were performed including instrumentation for recording of micro-magnetic parameters under superimposed tensile load [3, 4]. The samples were cylindrical, 80 mm length and 6 mm diameter. The samples were magnetized in the axial direction. The Barkhausen noise signals were excited by an alternating magnetic field which was applied to the sample by means of an electromagnet. The measurements parameters were: H_{max} = ±75A/cm and f_E = 0.05Hz. To acquire the Barkhausen noise signals and to suppress the influence of the exciting magnetic field, two air-coils were used in differential mode. The noise signals' induced voltage was recorded as a function of the tangential field strength, the resulting Barkhausen noise profile was then analyzed with regard to profile peaks and their associated magnetic field strengths. In order to record the $M_{MAX}(\sigma)$ curves 10 Barkhausen noise profile curves were cycled and averaged in order to enhance signal/noise-ratio [11].

Fig. 4: Experimental set-up diagram

5. Results and Discussions

In order to detect the MRS of IIIrd order Barkhausen noise measurements under superimposed tensile load stress on Fe-Cu samples were performed. Coherent Cu precipitates formed after the thermal ageing two different kinds of micro residual stresses in a ferritic matrix (see chapter 3).
In order to detect the MRS induced by the precipitation of the coherent Cu particles and to eliminate the influence of the compressive RS of Ist order induced by quenching and of the MRS of IInd order, induced by coherent Cu particles the micro magnetic measurements were performed in three steps (Fig. 5) as follows:

Step 1: Measurements after quenching – in that state the only influence on the micro magnetic measuring quantities is due to the compressive RS induced by quenching into water after the solution heat treatment temperature (Fig. 5, curve - 0min/500°C: A).

Step 2: Measurements after the thermal ageing, when coherent Cu particles form and induce micro residual stresses of IInd and IIIrd order (Fig. 5, curve - 390min/500°C: B).

Step 3: Measurements after the thermal ageing, when coherent Cu particles transform into incoherent Cu particles and therefore the MRS of IIIrd order disappear and the MRS of IInd order remain in the Fe matrix (Fig. 5, curve – 1500min/500°C: C).

Furthermore, in order to determine only the precipitation-induced MRS of IInd and IIIrd order and to eliminate the influence of the quenching, the shift ($\Delta\sigma$) between the $M_{MAX}(\sigma)$ curve measured after the thermal ageing and the $M_{MAX}(\sigma)$ curve measured after the quenching was calculated. That shift represents the precipitation-induced MRS of IInd and IIIrd order:

$$A - B = \text{MRS II}^{nd} + \text{MRS III}^{rd} \qquad \text{(Eq. 1)}$$

In order to determine the MRS of IIIrd order the MRS of IInd order have to be known. The shift between the $M_{MAX}(\sigma)$ curve corresponding to the as-quenched state and the $M_{MAX}(\sigma)$ curve corresponding to the second thermal aged state (1500min/500°C) represents the MRS of IInd order because after 1500 min at 500°C the Cu precipitates transform from coherent into incoherent particles and the MRS of IIIrd order disappear:

$$A - C = \text{MRS II}^{nd} \qquad \text{(Eq. 2)}$$

The measured shift between the $M_{MAX}(\sigma)$ curve after the first thermal ageing (390min/500°C) and the $M_{MAX}(\sigma)$ curve corresponding to the second thermal aged state (1500min/500°C) represents the MRS of IIIrd order:

$$\text{MRS III}^{rd} = (A - C) – (A - B) \qquad \text{(Eq. 3)}$$

$$\text{MRS III}^{rd} = B – C \qquad \text{(Eq. 4)}$$

Fig. 5: Experimentally determined tensile load stress dependence of the maximum Barkhausen noise amplitude (M_{MAX}) of a Fe-1.0%Cu-alloy for the as-quenched state, a thermal aged state containing only coherent Cu particles and a thermal aged state containing only incoherent Cu particles

As Eq. 4 shows in order to determine the precipitation induced MRS of IIIrd order it is necessary to measure the $M_{MAX}(\sigma)$ curve of an overaged state (Ostwald ripening) and the $M_{MAX}(\sigma)$ curve during the early stage of precipitation.

6. Conclusions

A testing method based on micro-magnetic measurement techniques was developed to determine quantitatively MRS of IIIrd order. The developed test method allows a quantitative determination of RS without the need for a reference method such as the X-ray RS measurement. Therefore, this testing technique opens a wide range of possible industrial applications provided that loading can be performed.

7. References

[1] Macherauch, E., Kloos, K. H.: Origin, Measurement and Evaluation of Residual Stresses, Residual Stresses in Science and Technology, DGM Informationsgesellschaft-Verlag, 1987

[2] Kneller, E.: Ferromagnetismus, Springer Verlag, 1962

[3] Altpeter, I., Becking R., Kern, R., Kröning, M., Hartmann, S.: Mikromagnetische Ermittlung von thermisch induzierten Eigenspannungen in Stählen und weißem Gusseisen, in: Deutsche Forschungsgemeinschaft, Eigenspannungen und Verzug durch Wärmeeinwirkung, Forschungsbericht, Wiley-Vch, 1999, 407/426

[4] Altpeter, I.; Dobmann, G.; Kröning, M.; Rabung, M.; Szielasko, K.: Micro-Magnetic Evaluation of Micro Residual Stresses of the IInd and IIIrd Order, NDT&E International. 42 (2009), 4, 283-290.

[5] Soisson, F., Barbu, A., Martin, G.: Monte Carlo Simulation of Copper Precipitation in Dilute Iron-Copper Alloys during Thermal Ageing and under Electron Irradiation, Acta Materialia 44, No. 9, 1996

[6] Nagai, Y., Hasegawa, M.: Positron confinement in ultrafine embedded particles: Quantum-dot-like state in an Fe-Cu alloy, Physical Review B, Vol. 61, No.10, 2000

[7] Goodman, S.R., Brenner, S.S., Low, J.R.: FIM-Atom Probe Study of the Precipitation of Copper from Iron-1.4 At.Pct.Copper, Metallurgical Transaction, Vol. 4, 1973

[8] Barbu, A., Mathon, M.H., Maury, F., Beneu, B., de Novion, C.H., Belliard, J.F.: A comparison of the effect of electron irradiation and thermal aging on the hardness of FeCu Binary alloys, Journal of Nuclear Materials, 1998

[9] Hornbogen, E.: Werkstoffe – Aufbau und Eigenschaften von Keramik-, Metall-, Polymer- und Verbundwerkstoffen, Springer-Verlag, 1994

[10] Rabung, M., Altpeter, Kizler P., Schmauder, S. : Ermittlung von Mikroeigenspannungen herrührend von kohärenten nanoskaligen Ausscheidungen, final report of the german research society (DFG) 2007

[11] Rabung, M., Altpeter, I., Dobmann, G., Szielasko, K.: MICRO-MAGNETIC EVALUATION OF MICRO-RESIDUAL STRESSES OF THE IIND AND IIIRD ORDER, Welding in the world N°06 Vol. 56 2012

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