Quantitative Non-Destructive Detection of Residual Stresses of the 2nd and 3rd Order by Using Micro Magnetic Methods

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Abstract. Micro residual stresses (MRS) of the 2nd and 3rd order play an important role in the lifetime analysis of thermally-cycled materials. The coherent residual stresses (MRS of 3rd order) appear when the lattice parameter of the second phase particles (coherently embedded in the matrix) and the lattice parameter of the matrix are different from each other. Such differences in case of the Fe-Cu alloys between the lattice parameters of coherent Cu precipitates and the lattice parameters of the α-Fe matrix lead to distortion of the lattice around the Cu particles causing coherent tensile MRS of 3rd order. Thermally induced MRS of the 2nd order appear when individual material phases exhibit different thermal expansion coefficients. Such differences in temperature-related expansion coefficients of copper precipitates and the α-Fe matrix promote the development of thermally induced compressive MRS of the 2nd order. The main objective of the presented research project is to develop an integral, economical and fast micro-magnetic non-destructive method for quantitative characterisation of MRS of the 2nd and 3rd order in Fe-Cu and Fe-Cu-Ni alloys. For this purpose Fe-Cu and Fe-Cu-Ni alloy samples with different Cu and Ni contents were prepared.

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 MRS are balanced in a mechanical equilibrium. Measurement of residual stresses in materials is of increasing interest. Generally speaking, residual stresses in superimposition with load induced stresses have advantages when they compensate. Therefore the knowledge of the spatial distribution of both residual stresses of second and third order is essential for the life management of the components. 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 micromagnetic ND techniques are suitable for the characterization of the residual stress state of the component.
The main objective of the present research work was to develop a micro-magnetic non-destructive method as an alternative to the radiographic method for quantitative characterisation of the microscopic residual stresses in multi-phase, iron-based materials, which is very time consuming.

2. Theoretical Background

2.1 Residual stresses in multi-phase materials

Residual stresses occur always if regions of the material are inhomogeneously elastically or plastically deformed in such a permanent manner that stress incompatibilities appear. The basic physical mechanisms for the build-up of residual stresses are a possible criteria to define the different types as: (1) residual stresses of 1st order, also called macro residual stresses, are nearly homogeneous across large areas of a material (several grains) and equilibrated within the whole body, (2) residual stresses of 2nd order are nearly homogeneous across microscopic areas of a material (one grain or parts of a grain), equilibrated across a sufficient number of grains and are nearly homogeneous, (3) residual stresses of 3rd order, are inhomogeneous across submicroscopic areas of a material (several atomic distances within a grain), equilibrated across small parts of a grain and are inhomogeneous [1]. The two last kinds of residual stresses are also called micro residual stresses

The Cu precipitates coherently embedded in a ferritic matrix induce two different kinds of MRS: coherence tensile MRS of IIIrd order and thermally-induced compressive MRS of IInd order. The coherence MRS of the IIIrd order appear when the lattice parameter of the IInd phase particles coherently embedded in the matrix and the lattice parameter of the matrix are different. The thermally-induced MRS of IInd order arise at the interface between different material phases because of their different thermal expansion coefficient. In case of the Fe-Cu system the bigger lattice parameter and thermal expansion coefficient of Cu compared to Fe lead to coherence tensile MRS of IIIrd order and thermally-induced compressive MRS of IInd order respectively.

2.2 Magnetic methods - Barkhausen noise measurements

Ferromagnetic materials consist of small, finite regions called domains, separated from each other by Bloch walls (BW). In the magnetic structure of a ferrous magnetic material only two kinds of BWs are observed: 180°-BWs are magnetostrictively inactive, have short range stress fields resulting in high mobility and sensitivity to external stress, and 90°-BWs are magnetostrictively active, have long-range stress fields and therefore low mobility. The BW movement takes place discontinuously because they are temporarily pinned by microstructural obstacles like dislocations, precipitates, phase or grains boundaries in a polycrystalline material. The stepwise pull-out of the walls from the obstacles changes the magnetisation state locally and is called Barkhausen event. These local magnetisation changes induce pulsed eddy currents into the sample and electrical voltage pulses into a pick-up coil near the surface, the so called Barkhausen noise [2].

2.3 Integral Load-Stress Related Barkhausen Noise

Smallest changes in the materials state e.g. the change of the MRS induced by the precipitation of coherent Cu particles sensitively affect the magnetic domain structure. The micro-magnetic test methods have a high potential to detect change of the MRS because they sensitively react to the changes of the Bloch wall (BW) configuration. The integral micro-magnetic concept used, is based on the measurements of the load-stress-dependent maximum Barkhausen noise amplitude (M_{MAX}). The maximum of the Barkhausen noise amplitude obtained during one hysteresis cycle is recorded as a function of the increasing load stress, the so called $M_{MAX}(\sigma)$-curve. These series of Barkhausen noise maxima 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 characterisation of residual stress variations 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 sooner their maximim, i.e. the curve shifts to the left-hand side (Fig. 1, red curve) and vice versa in case of the superimposed compressive stresses (Fig. 1, blue curve).

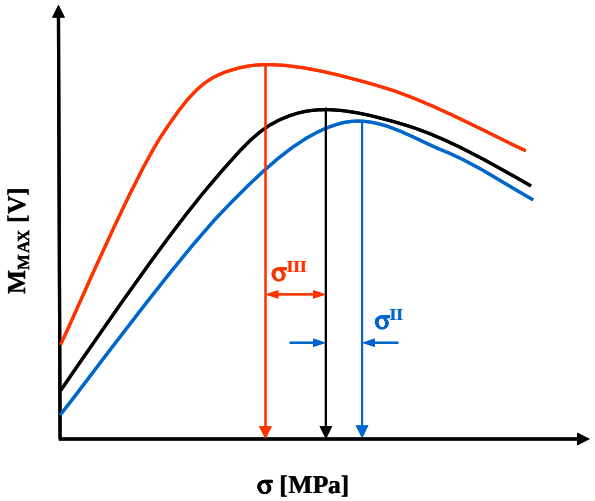

Fig. 1: Schematically represented shift of the tensile load stress dependence of the maximum Barkhausen noise amplitude ($M_{MAX}(\sigma)$-curve) for a higher tensile residual stress state (red curve) and for a higher compressive residual stress state (blue curve)

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 corresponding to the stress-free state 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 corresponding to the higher compressive MRS state back to the left-hand side along the load stress axis.
3) When finally, the $M_{MAX}(\sigma)$ curve is placed to the left-hand side compared to the stress free state, the tensile MRS are bigger than the compressive MRS and the value of that shift represent the difference between the absolute value of the tensile MRS and the absolute value of the compressive MRS.

3. Experimental setup

In order to determine the MRS Barkhausen noise measurements under superimposed tensile load stress were performed. The experimental set-up includes instrumentation for the recording of micro-magnetic parameters, like i. e M_{MAX} under tensile load. The samples are magnetised by an alternating magnetic field in the longitudinal direction by using an electromagnetic yoke. The noise signals were captured as an induced voltage by an induction coil surrounding the magnetised specimen and recorded as a function of the tangential field strength after suitable filtering, amplification and rectification. With the help of a modular measurement system, the resulting Barkhausen noise profile was then analysed with regard to profile peaks and their associated magnetic field strengths.

4. Investigated materials

For the present study five binary Fe-Cu alloys with Cu content from 0.3 to 1.7 wt.% and two ternary Fe-Cu-Ni alloys were manufactured and investigated. The samples were prepared from pure (99.99%) Fe-powder, pure (99.99%) Cu-powder and Ni-powder (99.99%) have been molten by arc melting under protective gas (Ar, 700 mbar) in rods casting moulds. These rods were annealed at 850°C for 2 hours, quenched into water and warm-rolled (900°C). In order to obtain precipitation hardened alloys, the typical heat treatment is: (1) solution annealing in the monophase area, (2) quenching and (3) thermal aging in the two-phase area [4-9]. During the thermal aging 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 whereas incoherent precipitates cause the decrease of the hardness. Because the goal of the present research work is the determination of the MRS induced only by the coherent Cu particles a test thermal treatment was performed in order to determine the exactly thermal ageing time up to the Cu precipitates are still coherent. Therefore on Fe-Cu and Fe-Cu-Ni test specimens a precipitation hardening heat treatment was applied as follows: (1) solution annealing at 850°C for 2 hours, (2) quenching into water and (3) thermal aging at 500°C up to the hardness maximum was reached. During the thermal ageing Vickers hardness measurements were stepwise performed. The mechanical hardness dependence of the thermal ageing time for each sample remained at the beginning for shorter or longer time constant and reached its maximum sooner or later respectivelly, function of Cu content. With increasing Cu content the hardness started to increase and reached its maximum was reached sooner. This is due to an increasing possibility of nucleation with rising Cu content, because the diffusion ways, which have to be crossed by the Cu atoms for Cu precipitates' nucleation are shorter. In order to obtain Fe-Cu alloys containing small, coherent Cu precipitates, the thermal aging time for each sample must be shorter than the thermal aging time corresponding to the hardness maximum.
As a result of this heat treatment test, samples for electromagnetic, small angle neutron scattering (SANS) and Vickers hardness measurements were manufactured, as follows: solution annealing at 850°C for 2 hours, (2) quenching into water and (3) thermal aging at 500°C so long as in the table 1 is showed. The heat treatment was applied in order to obtain Fe-Cu alloys with coherent Cu precipitates as only point defects. In order to study the influence of the variation of Ni content the Fe-Cu-Ni samples were so long thermal aged as the Fe-0.65 wt.% Cu sample.

Table 1: Duration of the thermal ageing at 500 °C of the Fe-Cu samples

Cu content [wt. %]	Thermal ageing time [h]
0.3	25
0.65	12
1.0	3.5
1.4	2.17
1.7	0.83

5. Results and Discussions

5.1 SANS measurements

Changes of the precipitation state, like variations of the particle radius, particles volume fraction or/and particles density yield to a change of the micro residual stress state. In order to determine the Cu precipitates size, volume fraction and their density Small Angle Neutron Scattering (SANS) measurements were performed. Those measurements were carried out with the instrument SANS-2 at the Geesthacht Neutron Facility (GeNF). The SANS measurements showed that the investigated Fe-Cu as well as Fe-Cu-Ni samples contain Cu particles with mean radius between 0.6 to 1.1 nm. It is well known that the Cu particles in the Fe-Cu system show a coherency with the surrounding Fe matrix when their radius is smaller than 5 nm. In that way by means of the SANS measurements it was proved, that the manufacturated samples only coherent Cu particles contain. In case of the Fe-Cu samples the Cu particles density and volume fraction increase with increasing Cu content (table 2). The SANS measurements performed on the Fe-Cu-Ni samples showed that Ni caused an obstruction of the precipitation, because an increase of Ni content results in a decrease of the Cu precipitates density (table 2).

Table 2: SANS measurements on Fe-Cu samples

Cu content [wt.%]	**Ni content [wt.%]**	**Radius [nm]**	**Volume fraction [%]**	**Density [cm^{-3}]**
0.3	0	0.733	0.0188	$5.93x10^{16}$
0.65	0	0.81	0.111	$2.27x10^{17}$
1.0	0	0.646	0.347	$9.35x10^{17}$
1.4	0	1.17	0.729	$9.26x10^{17}$
1.7	0	1.06	0.676	$1.23x10^{18}$
0.65	1.0	1.1	0.152	$1.79x10^{17}$
0.65	1.3	1.03	0.119	$1.66x10^{17}$

5.2 Mechanical hardness measurements

Changes in the Cu precipitation state cause changes in the mechanical hardness. The precipitation of coherent Cu particles causes an increase of the mechanical hardness of about 70 HV 5 with rising Cu content (Fig. 2). Measurements of the mechanical hardness on both kinds of alloys showed that during the early stages of the thermal ageing the hardness remains constant. That fact is conforming to the theory, which indicates that during that stage the nucleation of the Cu particles takes place.
Measurements of the mechanical hardness on the Fe-Cu-Ni samples showed that an increase of the Ni content caused a decrease of the mechanical hardness. A possible explanation is that an increase of the Ni content causes a stronger impede of the nucleation of the Cu particles, that can be well correlated with the SANS results which showed a decrease of the particles density (table 2).

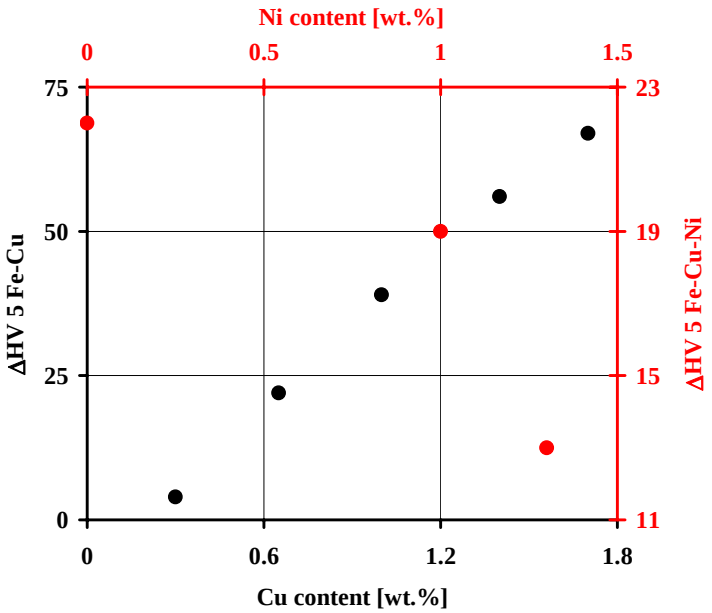

Fig. 2: Increase of the mechanical hardness caused by the increase of the Cu content and of Ni content

5.3 Micro magnetic measurements

To establish load-stress related Barkhausen noise measurements, a measurement system was installed to record the M_{MAX} values during simultaneous tensile loading. In order to detect the precipitation-induced change of MRS, Barkhausen noise measurements under superimposed tensile load stress were performed. By means of that micro-magnetic method a superimposition of both kinds of residual stresses is measured. In order to separate the coherent MRS of IIIrd order from the thermally-induced MRS of IInd order in Fe-Cu based alloys measurements at a temperature should be performed where the thermal expansion coefficient of Cu and Fe are identical. That requirement is fulfilled only above 1000 °C. But above 1000 °C the Fe-Cu based alloys containing Cu precipitates are monophase materials and the experiment offers no further informations. Therefore MRS of IInd and of IIIrd order cannot be separately evaluated.

In order to detect the MRS induced by the precipitation of the coherent Cu particles and to eliminate the influence of the compressive residual stresses induced by quenching, the micro magnetic measurements were performed in three steps (Fig. 3) as follows:

1) Measurements after quenching – in that state the only one influence on the micro magnetic measuring quantities is due to the compressive residual stresses induced by quenching from the solution heat treatment temperature (850 °C) into water (Fig. 3, black curve – 0 min).
2) Measurements at the beginning of the increase of the mechanical hardness, this means after the nucleation of Cu particles. During that stage the heating at 500 °C causes a decrease of the compressive residual stresses induced by quenching (Fig. 3, red curve – 5min).

Measurements after the thermal ageing, when the coherent Cu particles form and the induced MRS occur (Fig. 3, green curve – 50 min).

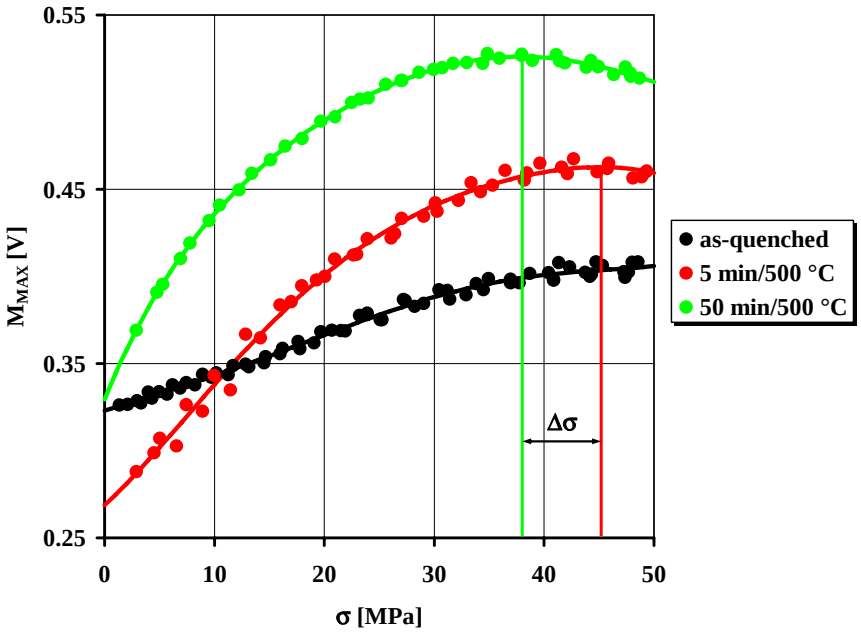

Fig. 3: Experimentally determined tensile load stress dependence of the maximum Barkhausen noise amplitude (M_{MAX}) for the Fe-1.7 wt.% Cu sample

As the mechanical hardness measurements indicated, at the beginning of the thermal ageing, no changes occurred. This suggests that only after that stage of the thermal ageing the precipitation-induced MRS occur. Further on in order to determine only the precipitation-induced MRS and to eliminate the influence of the quenching and the initial stage of the thermal ageing, the shift ($\Delta\sigma$) between the curve measured after the thermal ageing and the curve measured after the initial stage of the thermal ageing was calculated. That shift between the $M_{MAX}(\sigma)$ curve after the thermal ageing and the $M_{MAX}(\sigma)$ curve after the nucleation to the left-hand side of the tensile load stress axis represents the precipitation-induced MRS.

The micro magnetic measurements showed that the $M_{MAX}(\sigma)$ curves for the thermal-aged states of the Fe-Cu samples as well as of the Fe-Cu-Ni samples shift to the left-hand side of the tensile load stress axis compared to the $M_{MAX}(\sigma)$ curves (Fig. 3). Due to the fact that an increase of the tensile residual stresses causes a shift of the $M_{MAX}(\sigma)$ curve to the left-hand side of the tensile load stress axis and vice versa in case of the increasing superimposed compressive stresses (Fig. 1), it can be concluded that in case of the investigated samples the influence of the coherence tensile MRS preponderates compared with the thermally-induced compressive residual stresses. In conformity with the Fig 1 the micro magnetic determination of the micro residual stress state can be resumed as follow: the measured shift represents the difference between the coherence tensile MRS and the thermally-induced compressive MRS. This means that the coherence tensile MRS exceed the thermally-induced compressive MRS with that measured amount ($\Delta\sigma$).

The shift between both curves corresponding to the thermal-aged state and the nucleation as function of the Cu content increases with increasing Cu content (Fig. 4).

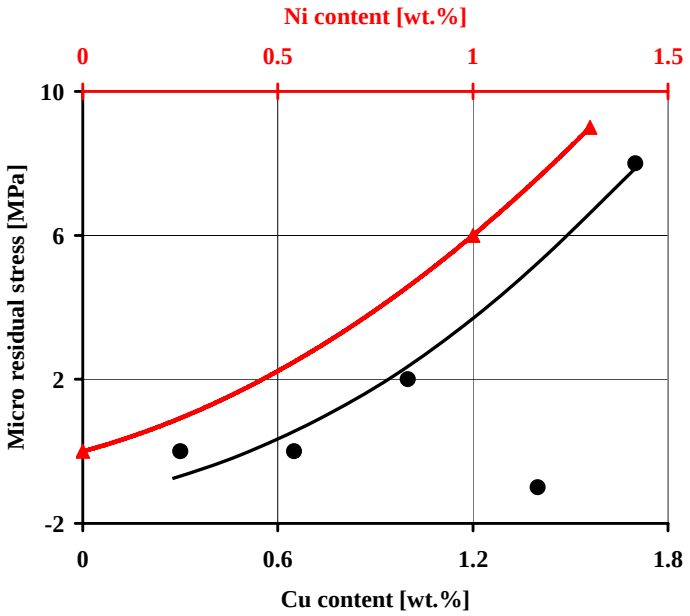

Fig. 4: Micro residual stress values in the Fe-Cu and Fe-Cu-Ni samples as function of the Cu content and Ni content respectively

The measurements on the sample Fe-1.4 wt.% Cu showed that $\Delta\sigma$ is negative which suggested that the thermally induced compressive MRS are higher than the coherence tensile MRS. In case of that sample the volume fraction is higher than in case of the sample with higher Cu content, but the density of the particles is smaller. That fact yields to the conclusion that the thermally induced compressive residual stresses are influenced preponderantly by the variation of the volume fraction and the coherence tensile MRS are influenced by the variation of the density of the particles.
In case of the Fe-Cu-Ni samples an increase of the Ni content yields also to an increase of the coherence tensile MRS, though the particles density decreases. A possible explanation is that the interface between matrix and particles is inhomogeneous because it contains also Ni atoms, which yields to a change of the micro residual stress state at the interface. The Ni atoms have an influence due to its thermal expansion coefficient on the thermally-induced compressive MRS and due to the different lattice parameter compared to Fe and Cu they have an influence on the coherence tensile MRS. But information about the interface between the matrix and particles in the Fe-Cu-Ni samples are expected from Atom Probe Field Ion Microscopy (APFIM) of which measurements are planned.

6. Conclusions

A testing method to characterise quantitatively MRS was developed. This method is based on tensile-load-dependent Barkhausen noise measurements, where the peaks of the Barkhausen noise amplitude are recorded as a function of the increasing tensile stresses. The shift of the $M_{MAX}(\sigma)$ curves parallel to the stress axis is a measure for RS originating from the second phase. The developed test method allows for 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 so far loading can be performed.

7. References

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