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RESEARCH ARTICLE

Changes of the Magnetic Properties during Heat Treatment in AISI 304

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Abstract
The aim of this study was to evaluate changes of the magnetic properties during heat treatment in austenitic stainless steel AISI 304. Investigated changes were caused by plastic deformation in material. Specific composition of alloying elements in austenitic stainless steels has got influence on their deformation behavior. Samples were heat treated before measurement of magnetic properties in different intervals of temperatures. The first series of measurements has shown, that it is necessary to make more precise analysis of temperature intervals in the second part of experiment. For verification of structural changes in material there was used hardness test Vickers method and observation by light optical microscope. There was proven that AISI 304 is slightly magnetic after cold forming, although in general it is considered as non-magnetic.

Keywords
stainless steel, austenite, martensite, magnetic properties

1 Introduction

Transportation is part of most people's everyday life. It is used for movement of people or goods between locations. The number of produced cars has increased significantly in last decades, so it is important to find out innovative technologies and practices. Automotive industry is one of the world's most important economic sectors. Energy saving and safety are nowadays the main goals for auto-making industry. High strength steels are used to reduce the weight of the vehicle and thus energy consumption (Zhu, 2012). The other goals are improvement of utility properties, comfort for driver and the passengers, design of vehicles etc.

The field of study in this article is change of the magnetic properties during heat treatment in austenitic stainless steel AISI 304. Stainless steels are used in automotive as motor vehicle applications because they are resistant to corrosion and high temperature oxidation, offer energy absorption properties and maintain their mechanical properties over a wide temperature range. A motor vehicle application of AISI 304 contains fuel tanks, exhaust systems, housing for catalytic converters and turborchargers. Other applications are chassis for buses and trucks, handrails, internal and external trim (bumpers, door scuff plates, headling bezels etc.), luggage racks and structural components.

The austenitic stainless steels are ternary alloys of Fe-Cr-Ni. They contain high percentage of Cr and Ni, but also other elements such as Mb, Ti, Al, Nb, Cu, N, S, P and Se may be added to increase corrosion resistance. Selection of added elements depends on required properties of a final product and specific environments, too. Their microstructures consist of very clean FCC crystals in which all alloying elements are held in solid solution. These steels are called austenitic because of their final structure. It is austenitic at room temperature. The austenitic stainless steels are applied in chemical, petrochemical, nuclear industry, architecture, food processing and many others.

The most widely used austenitic stainless steel is AISI 304. This material belongs to the type called high-alloy TRIP steels. These types of steels contain substantial number of alloying elements such as Cr and Ni, which improve pitting and corrosion resistance (Rodríguez-Martínez et al., 2011). It contains essentially 18% Cr and 8% Ni. Content of C is limited to maximum

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of 0,08% (Tourki et al., 2005). Mentioned steel is non-magnetic and it has got a cubic closed γ-phase. After plastic deformation, the phase is transformed to a BCC α'-martensite phase. The steel becomes ferromagnetic after plastic deformation. Many reports refer, that the magnetic effects of martensite content in AISI 304 is caused by progressive cold rolling (Yamasaki et al., 1996; Vértesy et al., 2005).

Effective tools to monitor the influence of the deformation behavior are magnetic techniques. Magnetic measurements are suitable for non-destructive testing, detection and characterization of modification and defects in materials (Vértesy et al., 2005). These techniques include measurement of magnetic hysteresis loop and magnetic Barkhausen emissions measurement (Mitra et al., 2006). A hysteresis loop shows the relationship between the induced magnetic flux density and the magnetizing force. The loop is generated by measuring the magnetic flux of a ferromagnetic material while the magnetizing force is changed. From hysteresis loop we can determine several primary magnetic properties of the material, such as retentivity, residual magnetism, permeability etc. The Barkhausen noise means that when an external magnetizing field through the ferromagnetic material is changed, the magnetization of material changes in series of discontinuous changes. It causes jumps in the magnetic flux through the material. These jumps of magnetization are interpreted as discrete changes in a size or a rotation of ferromagnetic domains. The jumps can be detected by winding a coil of wire around the bar, which is attached to an amplifier and a loudspeaker. When the amplifier produce sound in the loudspeaker, the unexpected transitions in the magnetization produce current pulses in a coil (O'Sullivan et al., 2004).

2 Experimental procedure

Experimental material for magnetic measurements was AISI 304. This austenitic stainless steel has got many interesting properties such as high ductility, excellent drawing, forming, and spinning properties. AISI 304 is essentially non-magnetic, but it becomes slightly magnetic when cold worked. Low carbon content means less carbide precipitation in the heat-affected zone during welding and a lower susceptibility to intergranular corrosion. It also resists to most oxidizing acids and salt spray. These properties make AISI 304 widely used stainless steel. The testing specimens were small blocks with dimensions 10 x 10 x 20 mm. The austenitic structure in mentioned steel is secured by balance between austenite-forming and ferrite-forming elements. Chemical composition of tested material is shown in Table 1. Detection of chemical composition was realized on spectrometer SpectromaxX.

The samples were heat-treated in furnace before measuring their magnetic properties. Letter (A) marked initial state of material. There were chosen values of temperatures at 100 °C (B), 400 °C (C), 800 °C (D), where the samples were heated for 15 minutes and temperature 1050 °C (E), where the sample was heated for 30 minutes. The last temperature was 700 °C (F). The sample was heated for 10 hours at this temperature. Comparison of magnetic properties is shown in Fig. 1. Measurement of magnetic properties in these intervals of temperatures showed that it was necessary to check interval between 400 °C and 800 °C more detailed. For the second measurement were chosen intervals of temperature 500 °C (G), 550 °C (H), 600 °C (I) and 620 °C (J). All of samples were heated for 15 minutes. The results of this measurement are stated in Fig. 2.

Table 1 Chemical composition of AISI 304

Element Material	**C**	**Cr**	**Ni**	**Mo**	**Mn**
AISI 304 [wt%]	0.05	19.59	7.84	0.49	1.57
Element Material	**Si**	**S**	**P**	**Ti**	**Fe**
AISI 304 [wt%]	0.29	0.03	0.05	-	ballance

There are many devices for measuring magnetic properties. This experiment was realized on device Magnet Physik, which is used for measuring of hysteresis loops. On this device, it is possible to determine magnetic quantities (remanence, coercivity), make measurements with surrounding coils to determine the magnetic mean values and measure at temperatures up to 200 °C. The measurement was performed under normal conditions at room temperature. The temperature of samples was 21 °C. The magnetic excitation fields that are necessary to record a hysteresis loop were generated by the electromagnet EP 3. The maximum current of the electromagnets power supply was set to ±10 A and time of increasing of current to maximum to 40 s. During all of the measurements the demagnetization was on.

Explanatory notes for Fig. 1 are listed in Table 2. At the temperature of 100 °C the investigated material has got a top of internal damping. In AISI 304 was observed one-way memory effect. In general, when a shape-memory material is in its cold state, the metal can be bent or stretched and will hold those shapes until heated above the transition temperature. Upon heating, the shape changes to its original. When the metal cools again it will remain in the hot shape, until deformed again. Martensite created from austenite by deformation is transformed back to austenite due to temperature. Shape memory effect is cancelled at the temperature of 400 °C. Heat treatment at the temperature of 800 °C involved recrystallization of investigated material. Recrystallization is the process in which deformed grains of the crystal structure are replaced by a new set of stress-free grains that nucleate and grow until all the original grains have been consumed. This process is used to eliminate all the effects of strain hardening such as heavy plastic deformation produced during cold working. Heating of material at 1050 °C for 30 minute caused solution annealing. The purpose of solution annealing is to dissolve any precipitates

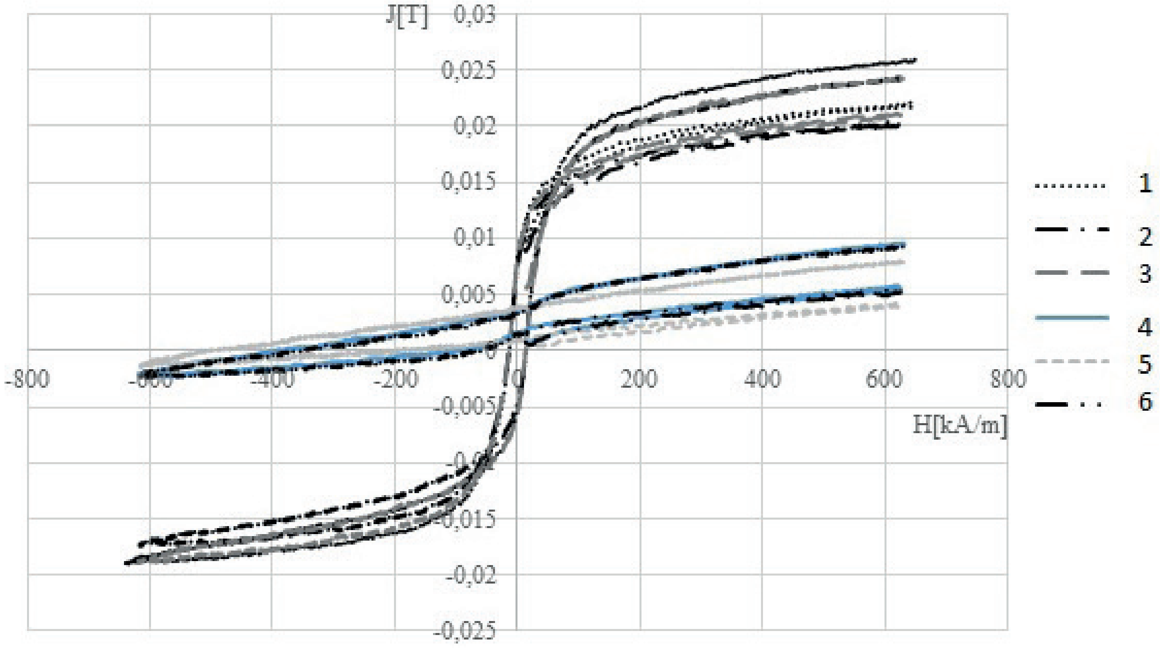

Fig. 1 Magnetic properties of AISI 304

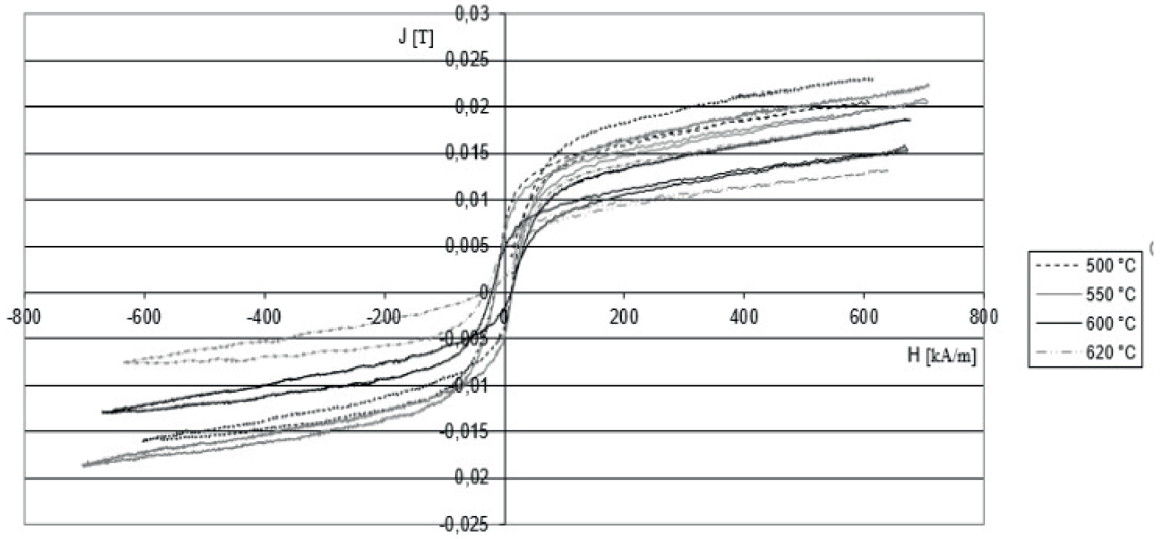

Fig. 2 Detailed comparison of magnetic properties in AISI 304

present in the material and transform the material at the solution annealing temperature into a single phase structure. At the end of the solution annealing process, the material is rapidly quenched down to room temperature to avoid any precipitation from occurring during cooling through lower temperature ranges where precipitates may form. The heat treatment at 700 °C for 10 hours causes sensitization of material. Sensitization is the loss of alloy integrity. It results from chromium depletion in the vicinity of carbides precipitated at grain boundaries. This causes the steel or alloy become susceptible to intergranular corrosion or intergranular stress corrosion cracking.

Table 2 Explanatory notes for Fig. 1

Number of curve	**1**	**2**
Temperature/Time	Initial state	100 °C/15 min
Number of curve	**3**	**4**
Temperature/Time	400 °C/15 min	800 °C/15 min
Number of curve	**5**	**6**
Temperature/Time	1050 °C/ 30 min	700 °C/10 hours

Differences in microstructure after heat treatment were observed by light optical microscope. In microstructure of AISI 304 in initial state were visible austenitic grains, α'-martensite

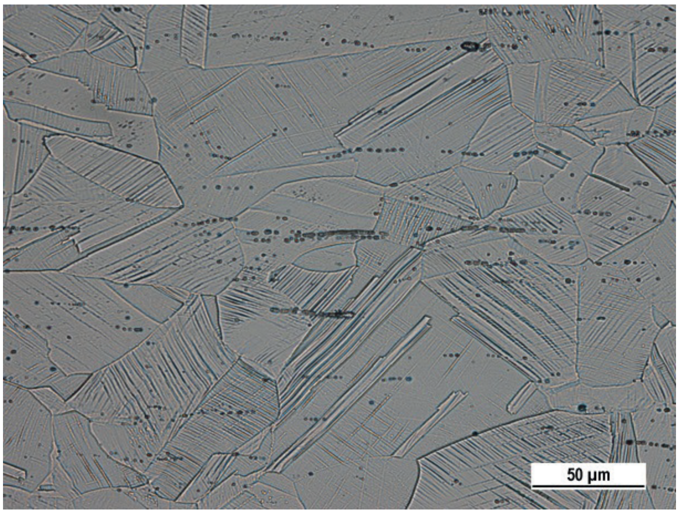

Fig. 3 Microstructure of AISI 304 in initial state

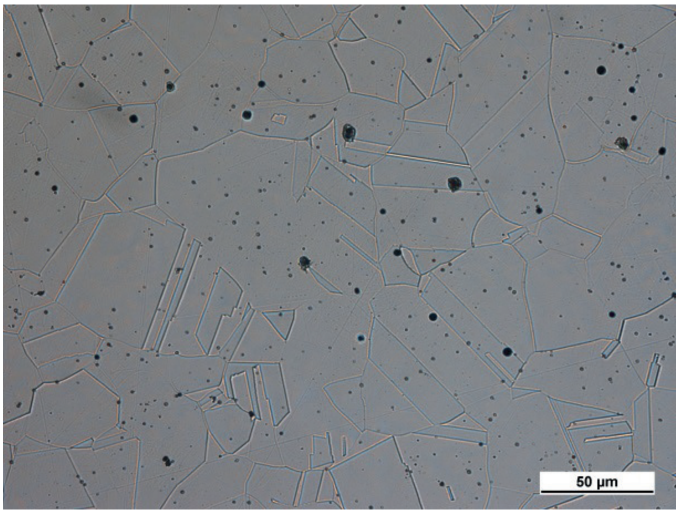

Fig. 4 Microstructure of AISI 304 after solution annealing

phase, deformation twins (Fig. 3). The structure has changed after solution annealing. There were mainly polyhedral austenitic grains and annealing twins (Fig. 4).

3 Results and discussions

Austenitic steels are in general paramagnetic materials, but AISI 304 showed magnetic behavior. It is caused by α'-martensite phase emergent by previous forming of material. The presence of α'-martensite phase was proven by checking of microstructure on light microscope (Fig. 3). Another factor that could affect magnetic behavior of austenitic steel is δ-ferrite. It causes that paramagnetic material turns its magnetic properties from paramagnetic to ferromagnetic, as mentioned in article (Martínková, 2010). However, the presence of δ-ferrite in initial status was not proven.

During experimental procedure it was necessary to choose suitable interval of temperatures for heat treatment before measurement of magnetic properties. Conventional austenitic steels are creep and corrosion resistant, but their use at elevated temperatures may be limited by the intergranular corrosion susceptibility that results from chromium depletion adjacent to grain boundaries due to carbide precipitation in the 450 °C - 850 °C range (Yae Kina et al., 2008).

In the first part of experiment this steel created hysteresis loop in the interval of temperatures from initial state to 400 °C. It means that material is ferromagnetic and contains α'-martensite phase caused by the plastic deformation. After heat-treatment at higher temperatures (about 700 °C) the curve did not connect at neither point, so did not create hysteresis loop. The steel has lost its magnetic properties. It is caused by structure elements decay. The α'-martensite phase is not contained in structure anymore (Fig. 4).

It was necessary to make more precise analysis of temperature intervals in the second part of experiment. In the first three steps we increased temperature of heat treatment from 500 °C by 50 °C for each sample. From temperature 600 °C we increased only by 20 °C to capture transition between magnetic properties. Fig. 2 shows, that increasing temperature changes magnetic properties of material. At the temperature 620 °C, the curve was close to x axis and the hysteresis loop was narrow. At higher temperatures the curve was not created.

For verification of structural changes in tested material was used hardness test Vickers method (Fig. 5). During the heat treatment the values of hardness decreased. After the recrystallization annealing of deformation reinforced steel, a gradual decrease in hardness occurred. Decrease of hardness was related with a gradual change of α'-martensite to austenite. The structure was gradually brought to equilibrium by heating. In the recrystallization annealing with a 15 minute time-out, there was a slight increase in the hardness value. It could be caused by refining austenitic grain. With longer durations at 850 °C, the hardness value again decreased. In this case, it may have occurred that longer holding time caused an increase and thickening of the austenitic grain. At the dissolution annealing temperature, the lowest hardness values were reached. There has been achieved homogenization of structure by rapid cooling to

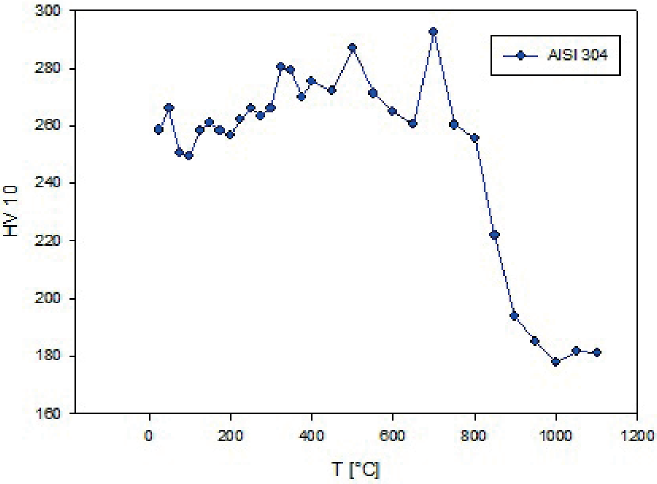

Fig. 5 Hardness test of AISI 304

water, thus there were no carbides, oxides and other inclusions produced. These structure elements would affect the resulting austenitic structure of the steel, if they were present. A similar case was also found in the publication written by Tukur et al., where the influence of temperature on mechanical properties, such as hardness, strength and ductility, was evaluated. Sensitized sample AISI 304 gave the highest hardness value (41 HRC) compared to the sample at baseline (36 HRC) and after annealing (20,4 HRC). Increased hardness was attributed to carbide formation along the grain boundaries of the sensitized specimen. The excluded carbides impeded the dislocation movement and reduced the deficiencies within the grit of the sensitized steel (Tukur et al., 2014).

The increase in hardness is associated with mechanical stiffness, i.e., plastic deformation results in deformation-induced martensite resulting from austenite transformation. The fact that part of the metastable austenite is transformed into deformation martensite is also caused, for example, by cold rolling, where plastic deformation is induced. Plastic deformation changes the properties of steel by increasing the density of dislocations which, in addition to its effect on mechanical properties, can help accelerate diffusion, phase transformation, and trigger the process of sensitization.

4 Summary and conclusions

We proved influence of plastic deformation on the magnetic properties of austenitic stainless steel by measurement of magnetic properties. It is caused by the presence of plastic deformation after previous forming. Cold forming caused the formation of α'- martensitic phase in the structure of austenitic stainless steel AISI 304. The α'- martensite is ferromagnetic, therefore tested material became slightly magnetic. Structural changes and presence of α'- martensite were verified by light optical microscope and also by hardness test Vickers method.

The measurement of magnetism has shown that magnetic properties of tested material changed with increasing temperature. The reason of this change is the structure elements decay. Issues of anomalous evolution of martensitic phase caused by heat treatment are more precise described in article written by Gauzzi et al., where the authors studied samples by high-temperature XRD during annealing in argon atmosphere at 400 °C for times up to 2.0×10^4 s. The experiments were performed by means of a high-temperature X-ray camera (Anton Paar HT 1600) using MoKα radiation ($\lambda = 0.71$ Å) (Gauzzi et al., 2006).

The measurement of magnetic properties is quick, contactless and non-destructive technique. It could be used for detection of damages in parts caused by intergranular corrosion. It is important to remark, that the distance to investigated surface affects the resulting values of magnetism.

As mentioned in article of Astudillo et al., the MBN (magnetic Barkhausen noise) technique detects accurately the micro structural changes caused by martensitic creation induced by stainless steel plastic deformation. In this research measurements performed with the micro durometer do not show significant differences between the 0º and 90º deformed samples. On the other hand, in all the tests, it was possible to establish the beginning of the martensitic deformation and its evolution by using dynamic registration of MBN (Astudillo et al., 2015).

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