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MAGNETIC CHARACTERISTICS OF ISOTHERMALLY AGED Cr-Ni-Mo-BASED ALLOYS WITH DIFFERENT δ-FERRITE CONTENTS

MAGNETNE LASTNOSTI IZOTERMNO ŽARJENIH ZLITIN Cr-Ni-Mo Z RAZLIČNO VSEBNOSTJO δ-FERITA

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We have investigated the influence of δ-ferrite content in Cr-Ni-Mo stainless-steel cast alloys on the magnetic characteristics. Samples of cast alloys with mass fractions 9-11 % of Ni, 18-21 % of Cr and 1.8-2.5 % of Mo, with three characteristically different δ-ferrite contents, were prepared with careful selection of appropriate amounts of alloying elements and a controlled solidification procedure. The samples were then aged in the operating temperature region (290 – 350 °C) for different periods of time (up to two years). The δ-ferrite content was determined with three different methods: i.e., empirically on the basis of chemical composition, magnetic-induction-based and metallographically. The magnetic-induction-based determination of δ-ferrite content showed that it does not change with ageing temperature and time. It confirms that only the internal ferrite structure is changed during ageing because of the spinodal decomposition.

The absolute magnetic properties were determined with a hysteresisgraph and a vibrating-sampling magnetometer (VSM). The results showed that the magnetic properties depend on the chemical composition (δ-ferrite content), the ageing temperature and the time. The chemical composition has the biggest influence, but the influence of the ageing temperature and time is insignificant and the scatter of the results is relatively large. The determination of the absolute magnetic properties is a destructive method and the mechanical preparation of the samples can influence the magnetic properties. Therefore, the method is not appropriate for the *in-situ* observation of the kinetics of spinodal decomposition. Measurements with the VSM are more appropriate than with the hysteresisgraph because much smaller samples with any geometry can be used.

Keywords: Cr-Ni-Mo based stainless steel, isothermal ageing, magnetic properties, influence of δ-ferrite

Raziskovali smo vpliv vsebnosti delta ferita na magnetne lastnosti Cr-Ni-Mo nerjavnih jeklenih litin. Izdelali smo tri različne litine v območju sestav z masnimi deleži med 9 in 11 % Ni, 18 in 21 % Cr in od 1,8 do 2,5 %Mo. Skrbno smo izbrali primerno vsebnost posameznih legirnih elementov in kontrolirali pogoje strjevanja, da smo dobili litine z različno, karakteristično vsebnostjo δ-ferita (s približno 4, 15 in 30 prostorninskimi deleži). Vzorce litin smo različno dolgo (do dveh let) izotermno žarili v temperaturnem območju (med 290 in 350 °C), ki je karakteristično za obratovanje uparjalnikov nuklearnih elektrarn. Vsebnost δ-ferita različno staranih vzorcev smo določevali na tri različne načine: empirično na osnovi kemijske analize, na osnovi merjenja magnetne indukcije in metalografsko. Meritve vsebnosti δ-ferita na metalografskih vzorcih so pokazale, da se le-ta ne spreminja s časom in temperaturo izotermnega žarjenja. To potrjuje predpostavko, da se med izotermnim žarjenjem ne spreminja vsebnost δ-ferita temveč le njegova notranja struktura zaradi spinodalnega razpada.

V vzorcih preiskovanih litin smo določili tudi absolutne magnetne lastnosti z merilnikom magnetne histereze in vibracijskim magnetometrom (VSM). Rezultati meritev so pokazali, da so magnetne lastnosti odvisne tako od sestave (vsebnosti δ-ferita) kot tudi temperature in časa izotermnega žarjenja. Največji vpliv ima vsebnost δ-ferita. Vpliv temperature in časa izotermnega žarjenja je manjši z opaznim raztrosom merjenih vrednosti. Določevanje magnetnih lastnosti je porušitvena metoda, saj moramo vzorec materiala izrezati iz elementa ali konstrukcije, ki jo preiskujemo. Mehanska izdelava preizkušanca lahko vpliva na magnetne lastnosti. Zato ta metoda ni najprimernejša za opazovanje sprememb *in-situ* med izotermnim žarjenjem oziroma spinodalnim razpadom. Magnetne meritve z VSM so primernejše, ker zahtevajo precej manjše vzorce poljubne oblike.

Ključne besede: nerjavna jekla na osnovi Cr-Ni-Mo, izotermno žarjenje, magnetne lastnosti, vpliv vsebnosti δ-ferita

1 INTRODUCTION

High-alloyed Cr-Ni-based stainless-steel cast alloys are frequently used in thermoelectric installations such as conventional and nuclear power plants (NPPs)[1,2]. Many years of exploitation of mechanical equipment in these objects have shown that the toughness of these alloys decreases with the operating time and temperature[3-7]. These alloys have a characteristic two-phase microstructure consisting of austenite and δ-ferrite[8–10]. The δ-ferrite content depends on the chemical composition of the alloy and on metallurgical factors: i.e., manufacturing technology and the exploitation conditions. Therefore, in alloys with a chemical composition the allowed ranges of content of alloying elements completely different microstructures can form. The result of this could be different mechanical properties of a material and it different behavior during its exploitation. Actually, our investigations have already confirmed that the Charpy impact energy and the resistance to stable crack growth of such alloys depend strongly on the δ-ferrite content[11,12].

Extensive investigations in the past indicate for the change of properties of these alloys the spinodal decomposition of δ-ferrite is responsible. Investigations also showed that the austenite phase does not play a

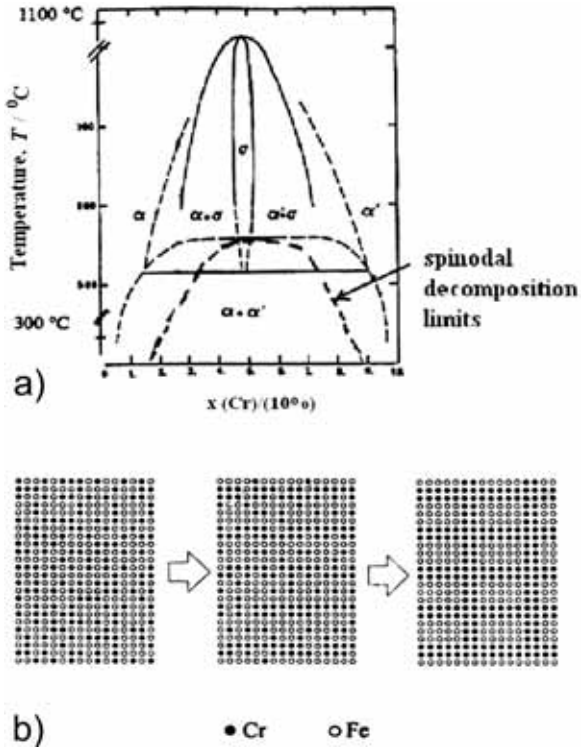

Figure 1: Schematic presentation of: a) quasibinary Fe-Cr-Ni-Mo phase diagram and b) redistribution of Fe and Cr atoms during spinodal decomposition[13]

Slika 1: Shematična predstavitev: a) kvazibinarni diagram Fe-Cr-Ni-Mo in b) prerazporeditev atomov Fe in Cr med spinodalnim razpadom[13]

significant part in this process. Spinodal decomposition in these types of alloys one can understand as it is schematically shown in **Figure 1**, and it is clear that the final result of spinodal decomposition is coherent, two-phase structure with different Cr-to-Ni ratios, even in a relatively low-temperature range (300 – 350 °C). This temperature range is similar to the operating-temperature range of vital parts of, for example, the coolant system[6]. The formation of two phases with the same crystal structure but different lattice parameters causes high internal elastic stresses, resulting in significant hardness increase and toughness decrease.

Besides the microstructural investigations, mechanical testing and analyses of isothermally aged materials[11,12] the aim of our work was also to find out if microstructural and related mechanical changes can be monitored and connected to a change of magnetic properties. One of the most useful non-destructive methods for the determination of δ-ferrite content is based on magnetic induction, because in two-phase systems (for example, ferrite-austenite) ferrite is the ferromagnetic phase. The amount of this phase is almost linearly proportional to the measured magnetic induction and therefore the method became a standard *in-situ* checking procedure for thermoelectric installations[9,14–15], especially when new reparative welds or parts are introduced during service. However, publications in the context of changes of the absolute magnetic properties (the hysteresis loops) of these types of alloys during isothermal annealing could not be found. Therefore, some investigations and analyses were performed relating to the change of the magnetic properties of thermally degraded material.

2 EXPERIMENTAL

The samples of a Cr-Ni-Mo-based cast alloy, CF-8M-type (ASTM A351), were prepared with a standard casting procedure. Twenty-kg batches of melts of the selected chemical composition were prepared in an inductive melting furnace and then cast into metal moulds under a controlled casting and solidification conditions. The solidification rate was controlled with the selection of an appropriate type of mould and melt/mould preheating. Cooled, cast ingots of each chemical composition (see **Table 1**), designated as alloys ***A***, ***B*** and ***C***, were cut and ground into parallel slices, approx. 10 mm thick. The actual chemical compositions of the prepared alloys were determined with optical and ion-coupled-plasma atomic emission spectroscopy (OES and ICP-AES).

On the basis of well-known empirical correlations (**equation (1)** and **(2)**) the Cr and Ni equivalents, as well as the δ-ferrite content were calculated[16]. Such a calculation shows that the alloy CF-8M inside the allowed chemical composition can practically contain from 0 to 90 volume fractions of δ-ferrite.

$$CR = \frac{w(Cr_{eq})}{w(Ni_{eq})} = \\ = \frac{w(Cr) + 1.5W(Si) + 1.4w(Mo) + w(Nb) - 4.99}{w(Ni) + 30w(C) + 0.5w(Mn) + 26(w(n) - 0.02) + 2.77} \quad (1)$$

$$F = -68.768 + 157.909CR - 133.171CR^2 + 47.1849CR^3 \quad (2)$$

Table 1: Nominal and actual chemical composition of the prepared alloys

Tabela 1: Nazivna in dejanske kemične sestave pripravljenih zlitin

Alloy designation	Chemical composition in mass fractions (w/%)								Cr and Ni equivalent	
	C	Si	Mn	P	S	Ni	Cr	Mo	Cr_{eq}	Ni_{eq}
CF-8M	<0.08	<2.0	<1.5	<0.04	<0.04	9–12	18–21	2-3	15.8–23.2	11.3–17.4
A	0.06	0.43	1.59	0.03	0.01	11.9	18.0	1.84	16.2	16.8
B	0.07	0.67	1.04	0.03	0.01	11.0	21.7	2.03	20.6	15.9
C	0.06	1.68	0.67	0.03	0.01	9.0	20.8	2.46	21.8	13.4

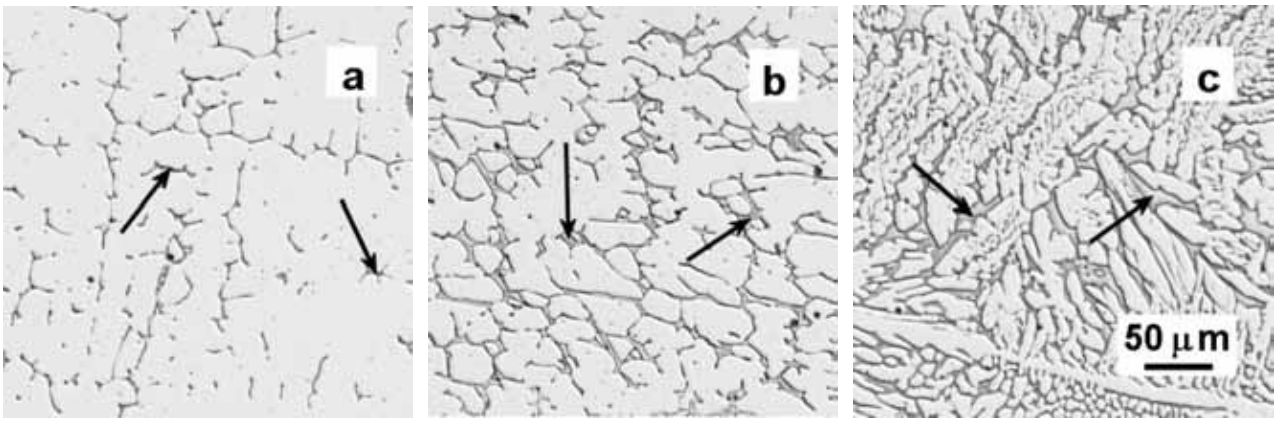

Figure 2: Micrographs of as-cast microstructures of investigated steels: **a)** alloy ***A*** with approx. 18Cr-12Ni, **b)** alloy ***B*** with 22Cr-11Ni and **c)** alloy ***C*** with approx 21Cr-9Ni, light-grey areas represent austenite, dark-grey areas marked with arrows are islands of δ-ferrite; LM, original magnification 200-times, selective etching of δ-ferrite (KOH : $K_3[Fe(CN)_6]$: H_2O=0.25 : 0.25 : 0.50).

Slika 2: Posnetki mikrostrukture preiskovanih jekel: **a)** zlitina ***A*** z 18 % Cr in 12 % Ni, **b)** zlitina ***B*** s približno 22 % Cr in 11 % Ni in **c)** zlitina ***C*** s približno 21 % Cr in 9 % Ni, svetla področja predstavljajo austenit, temna področja označena s puščicami pa so otočki δ-ferita; optični mikroskop, originalna povečava 200-krat, selektivno jedkanje na δ-ferit (KOH : $K_3[Fe(CN)_6]$: H_2O=0,25 : 0,25 : 0,50).

At the characteristic ingot positions the samples for metallographic investigations were cut and the average δ-ferrite content of each slice was determined magnetically and metallographically. Typical microstructures visible under a light microscope (LM) are presented in **Figures 2 a**, **b** and **c**.

A determination of the δ-ferrite content based on the magnetic induction (**Figure 3 a**) was performed with two instruments: i.e., an older one regularly used at NPPs and a new, digital one, the Feritscope MP-30 Fischer GmbH, Germany (**Figure 3 b**), with the appropriate calibration samples. The measured values obtained with different methods/instruments are shown in **Table 2**. It is clear that the differences in the measured values are relatively large. But the range of measured values obtained with one measuring method seems acceptable. The magnetically obtained values strongly depend on the sample surface and its shape, and therefore only the flat, polished surfaces of metallographic samples were used.

Slices of prepared alloys were then put into the laboratory batch furnaces and artificially aged (isothermally annealed) at elevated temperatures for different periods of time. The selected temperatures of ageing were 290, 320 and 350 °C and the time of 24 h, 1 and 6 months, and finally one and two years. After each ageing temperature/time cycle samples for microstructure characterization and mechanical testing were prepared. The average δ-ferrite content was again determined with an induction-based method on the polished metallographic samples.

Table 2: Initial δ-ferrite content, determined with different methods/ instruments

Tabela 2: Začetna vsebnost δ-ferita, določena na različne načine

Alloy designation	δ-ferrite content in volume fractions (φ/%)			
	Calculated*	Metallographically	Ferritmesser (NPP)	Ferritescope MP-30
A	2.1	1.2–6.3	1.5–2.5	3.5–4.7
B	14.9	14.0–18.3	10.0–12.0	14.6–19.2
C	38.8	28.5–34.0	26.0–28.0	27.9–32.9

* equations **(1)** and **(2)**

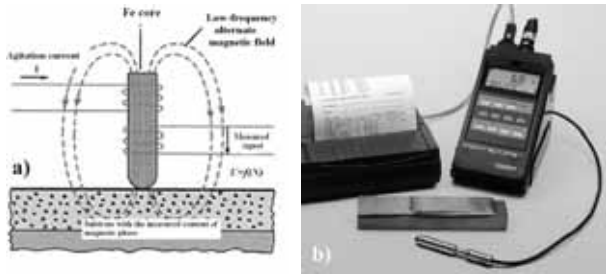

Figure 3: Determination of δ-ferrite content based on the magnetic induction: a) schematic presentation and b) modern digital ferritemeter (Ferritescope MP-30, Fischer GmbH, Germany)[17]

Slika 3: Določevanje vsebnosti δ-ferita na osnovi magnetne indukcije: a) shematični prikaz in b) moderen digitalni feritmeter (Ferritescope MP-30, Fischer GmbH, Nemčija)[17]

For the determination of the absolute magnetic properties with a hysteresisgraph (permagraph RE3, Magnet Physic Dr. Steingroever) cylinders dimensions of (ϕ 10 × 12) mm were used. For measurements with a vibrating-sampling magnetometer (VSM) small plates with dimensions of approx. (1.5 × 1.5 × 1.5) mm were used. The VSM has become a widely used instrument for determining the magnetic properties of a large variety of materials in the subzero and elevated temperature regions[18]. The investigated material was placed into a uniform magnetic field and mechanically vibrated, resulting in some magnetic flux change. The induced voltage in the pick-up coils is proportional to the magnetic moment of the sample.

3 RESULTS AND DISCUSSION

Figure 4 shows the results of the δ-ferrite content determined with Ferritescope, depending on the temperature and the time of isothermal ageing of the prepared alloys. It is clear that the δ-ferrite content is virtually independent of the ageing time and temperature. A slight trend for a decrease in the δ-ferrite content can be noticed, but only for alloy *C* at all temperatures and for alloy *B* at the highest temperature (350 °C). But it is in the range of the scatter of the measured results. Therefore, we can conclude that the induction-based method is not an appropriate method for monitoring of the kinetics of thermal degradation of the selected material. It seems an appropriate result and conclusion because during spinodal decomposition the changes occur at the nano-level, connected with small fluctuations of chemical composition and the cell parameters. However, the Ferritscope detects the average magnetic induction of much larger local regions (approx. 1 mm^2 and 1 mm^3, respectively), made up of ferromagnetic (ferrite) and nonmagnetic (austenite) grains. The method can, therefore, only serve as a control for the initial content of the chemical composition (δ-ferrite content), as well as an *in-situ* checking procedure for new welds and the replaced structural elements of thermoelectric installations during maintenance. As an example, **Figure 5** shows the change in δ-ferrite content across the but-weld of thin plates made of 316 L stainless steel at two different locations.

The hysteresis loop is the basic magnetic characteristic for ferromagnetic materials. From such a loop it is possible to read the remanence B_r, the coercivity H_c, the saturation magnetization B_s and the permeability μ. These properties are composition and structure sensitive; therefore, one can expect that it would be possible to monitor the kinetics of the spinodal decomposition.

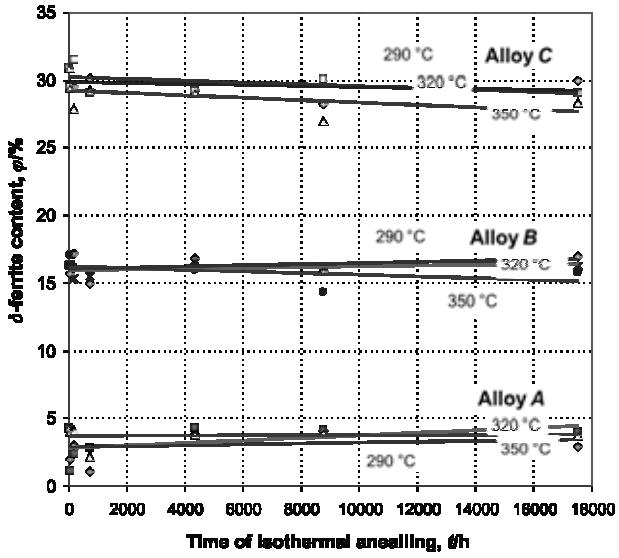

Figure 4: Average δ-ferrite content vs. time and temperature for isothermal annealing of all the prepared alloys, determined with the Feritscope MP-30 (Fischer GmbH, Germany)

Slika 4: Povprečna vsebnost δ-ferita v zlitinah *A*, *B* in *C* v odvisnosti od časa in temperature izotermnega žarjenja, določena s feritmetrom (Feritscope MP-30, Fischer GmbH, Nemčija)

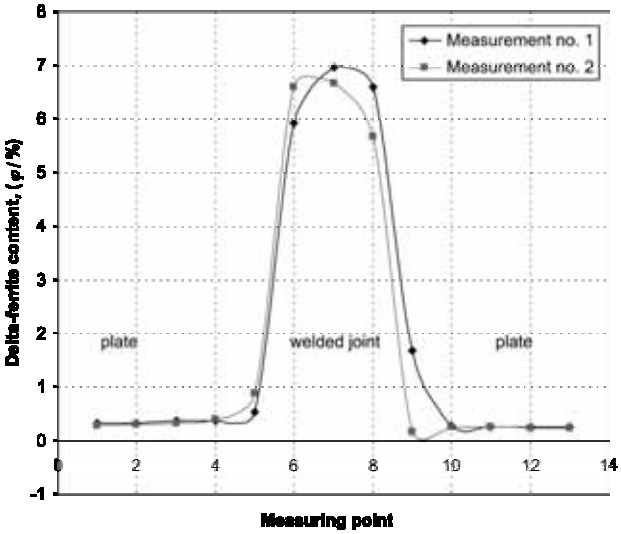

Figure 5: Change of δ-ferrite content across but-welded joint of thin plates made of 316 L stainless steel at two different measuring locations

Slika 5: Sprememba vsebnosti δ-ferita vzdolž zvarnega spoja dveh tankih plošč iz nerjavnega jekla vrste 316 L, merjena na dveh različnih lokacijah

The investigated material can be treated as a composite material with a different volume content of magnetic phase (δ-ferrite). Correspondingly, it is expected that the hysteresis curves of the investigated alloys change size and shape with an increased content of δ-ferrite. **Figure 6** shows the hysteresis loops of the original non-aged state of all three prepared alloys. As expected, alloy *C,* with the largest content of magnetic phase, has the narrowest and most upright hysteresis curve, with higher B_r, B_s and lower H_c in comparison

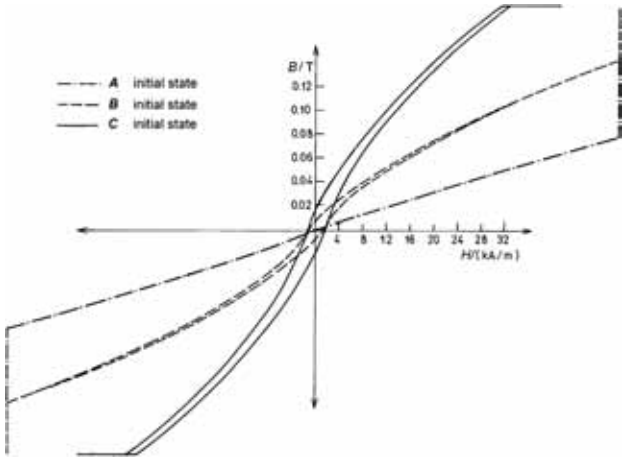

Figure 6: Hysteresis loops determined by Remagraph RE3, sample dimensions (ϕ 10 × 12) mm, original non-aged state of material

Slika 6: Histerezne zanke, ugotovljene z napravo Remagraph RE3, dimenzije vzorcev (ϕ 10 × 12) mm, originalno nežarjeno stanje materiala

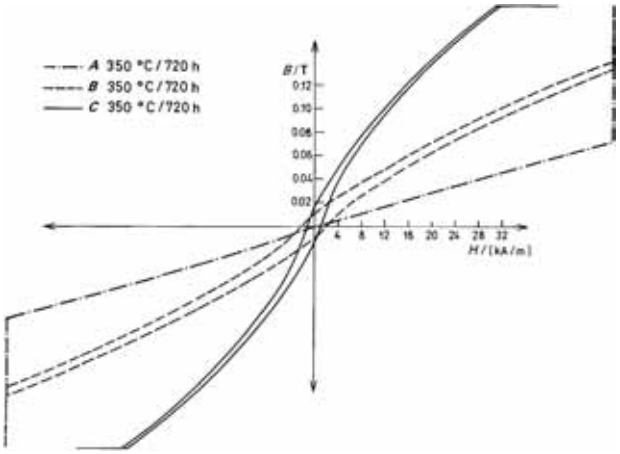

Figure 7: Hysteresis loops determined by Remagraph RE3, sample dimensions (ƒ 10 × 12) mm, aged 720 h at 350 °C

Slika 7: Histerezne zanke, ugotovljene z napravo Remagraph RE3, dimenzije vzorcev (ƒ 10 × 12) mm, izotermno žarjeno 720 h pri 350 °C

with alloy *B,* which has a lower content of ferromagnetic phase. Alloy *A* has a very low content of ferromagnetic phase and therefore shows an almost linear relation for the applied field vs. the induction (paramagnetic). **Figure 7** shows the hysteresis loops for alloys aged at 350 °C for 720 h. Its shapes and sizes are similar, and the differences in the magnetic properties compared to the original non-aged alloys are negligible. Therefore, the selected instrument is not the most appropriate for monitoring the spinodal decomposition of the selected alloys.

The hysteresis loops were then determined with the VSM. This method enables the use of smaller samples without any limits in terms of geometry and surface quality. The magnetic properties of the prepared materials were measured at 20 °C and 300 °C. The influence

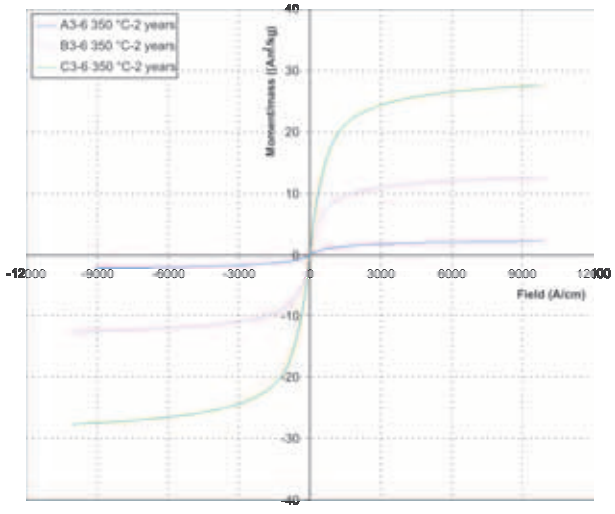

Figure 8: Typical hysteresis loops determined with the VSM, obtained for alloys *A*, *B* and *C* and aged 2 years at 350 °C

Slika 8: Tipične histerezne zanke, ugotovljene z VSM na zlitinah *A*, *B* in *C*, izotermno žarjene dve leti pri 350 °C

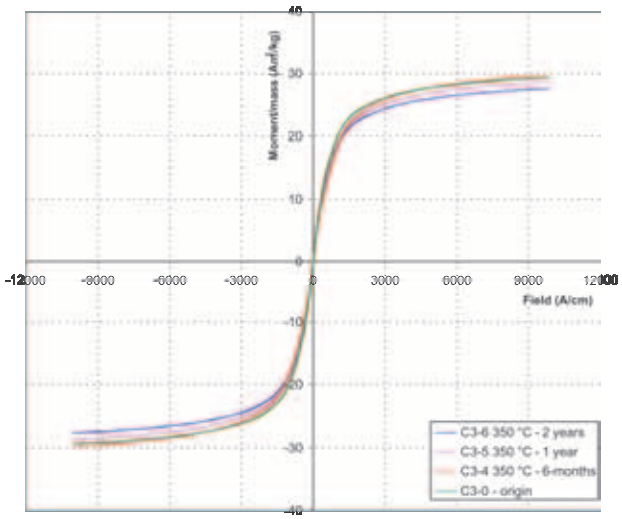

Figure 9: Typical hysteresis curves obtained with the VSM for alloy *C,* aged at 350 °C for different periods of time

Slika 9: Tipične histerezne zanke, ugotovljene z VSM pri zlitini *C*, različno dolgo časa izotermno žarjeni pri 350 °C

of the internal stresses had to be taken into account and any cutting/mechanical machining must be avoided or carefully carried out. **Figure 8** shows the room-temperature hysteresis loops of alloys aged at 350 °C for two years. The differences in the hysteresis curves are evident and they are similar to those obtained with the permagraph.

Figure 9 shows the room-temperature hysteresis loops of alloy ***C*** aged at 350 °C for different periods of time. It is clear that with increased ageing time the saturation magnetization decreases, but the changes are relatively small (approx. 27–29 emu/g). Also, the changes in the coercivity and the remanent induction are

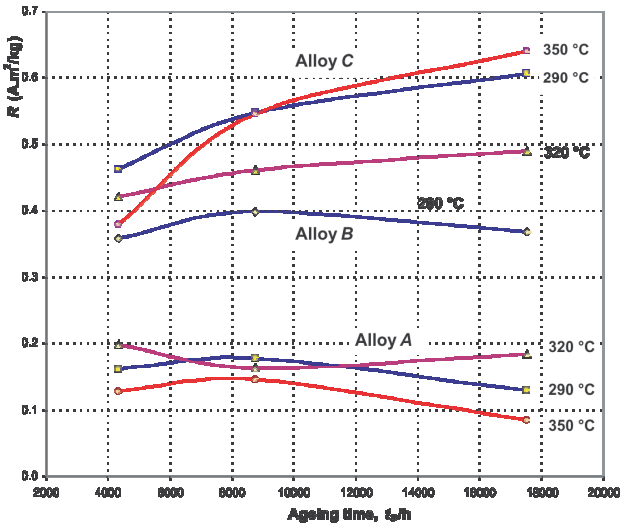

Figure 10: Average room-temperature remanent induction *R* vs. ageing time obtained for cast alloys with two different δ-ferrite content, aged at (290, 320 and 350) °C

Slika 10: Povprečna remanentna indukcija *R* določena pri 20 °C v odvisnosti od časa izotermnega žarjenja zlitin z različno vsebnostjo δ-ferita, žarjeno pri (290, 320 in 350) °C

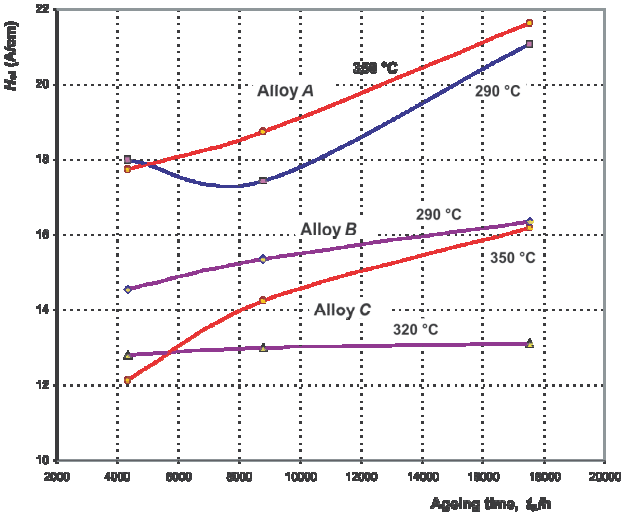

Figure 11: Average room-temperature coercivity vs. ageing time obtained for the investigated cast alloys, aged at (290, 320 and 350) °C

Slika 11: Povprečna koercitivnost, izmerjena pri 20 °C, v odvisnosti od časa izotermnega žarjenja zlitin z različno vsebnostjo δ-ferita, žarjeno pri (290, 320 in 350) °C.

very small and are close to the limits of the sensitivity of the VSM. The remanent induction (***R***) and the coercivity (H_{ci}) were read from the measured hysteresis curves and put into the diagrams in order to show the intrinsic properties vs. the ageing conditions for the individual alloys.

Figure 10 shows the room-temperature *R* of the alloys ***A***, ***B*** and ***C,*** aged up to two years at (290, 320 and 350) °C. Its change for non-magnetic alloy ***A*** is almost negligible, but it increases significantly for alloy ***C***, which has the highest content of ferromagnetic phase. The alloy ***B*** shows an almost negligible change, similar to alloy ***A***.

Figure 11 shows the room-temperature coercivity of alloys ***A***, ***B*** and ***C*** aged up to two years at selected temperatures. In this case the alloys show an increase of coercivity with ageing time. Surprisingly, alloy ***C*** at 320 °C does not show this increase.

Inconsistencies are noticed in the obtained results and shown in **Figures 10** and **11**. However, it is obvious that the change in the remanent induction and the coercivity are connected with structural changes. Therefore, we can also conclude that the kinetics of the spinodal decomposition can be monitored from the change in the absolute magnetic properties, but probably a more suitable instrument, specially designed for measurements of low coercivity soft magnetic materials, should be used.

4 CONCLUSIONS

The magnetic-induction-based determination of the δ-ferrite content showed that it does not change significantly with ageing temperature and time. It confirms that only the internal ferrite structure is changed, during ageing, because of the spinodal decomposition. Therefore, it can be concluded that magnetic induction is not appropriate method to observe spinodal decomposition in SS cast alloys. It can only serve as information or control for either the initial materials' content of δ-ferrite or as checking procedure during the maintenance of thermo-energetic objects.

The kinetics of spinodal decomposition can be monitored by a change in the absolute magnetic properties. However, these methods are destructive, as well as stress and temperature sensitive. The scatter of the results is relatively large and, therefore, sometimes they are inconsistent. The changes in the magnetic properties are also relatively small and so an appropriate measuring instrument has to be chosen.

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