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# The influence of heating temperature on coercive force and hardness changes in carbon hypoeutectoid steels

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#### **Keywords:**

magnetic properties, coercive force, temper colors, cooling in calm air, temperature interval Abstract. Hypoeutectoid steel with 0,25% carbon without preliminary heat treatment was investigated. The article describes an experimental study of the hardness and coercive force of this steel during heating and subsequent cooling in calm air. The magnetization depth of the device used and the penetration depth of the indenter when measuring hardness are significantly greater than the thickness of the total oxide films, therefore the measurement result is a complex value depending on the properties of the base metal and oxides. The influence on the studied parameters is proved not only of the structure of the base metal, but also of the properties of oxide films that appear on the steel surface in an oxygen-containing medium during heating. As a result, hardness and coercive force do not correlate with each other at all temperature intervals of heating. It is shown that the visual assessment of temperature by temper colors is subjective, and when the temperature threshold exceeds 500°C (for the research steel in given modes), the visually determined dependence between the temperature and the color of the sample surface after temperature exposure disappears.

## For citation

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# Влияние температуры нагрева на изменение коэрцитивной силы и твердости углеродистых доэвтектоидных сталей

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История статьи Поступила в редакцию: 25 января 2022 г. Доработана: 17 апреля 2022 г. Принята к публикации: 22 апреля 2022 г. Аннотация. Исследована доэвтектоидная сталь с содержанием углерода 0,25 % без предварительной термической обработки. Описано экспериментальное исследование твердости и коэрцитивной силы при нагреве и последующем охлаждении на спокойном воздухе. Глубина

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#### Ключевые слова:

магнитные свойства, коэрцитивная сила, цвета побежалости, охлаждение на спокойном воздухе, температурный интервал намагничивания используемого прибора и глубина проникновения индентора при измерении твердости значительно больше толщины суммарных оксидных пленок, поэтому результат измерения представляет собой комплексную величину, зависящую от свойств основного металла и оксидов. Доказано влияние на исследуемые параметры не только структуры основного металла, но и свойств оксидных пленок, возникающих на поверхности стали в кислородсодержащей среде при нагреве. В результате твердость и коэрцитивная сила не коррелируют друг с другом во всех температурных интервалах нагрева. Показано, что визуальная оценка температуры по цветам побежалости носит субъективный характер и при превышении температурного порога в 500 °C (для исследуемой стали на заданных режимах) визуально определяемая зависимость между температурой и цветом поверхности образца после температурного воздействия исчезает.

#### Для цитирования

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#### Introduction

When a temperature load is applied and subsequent cooling occurs in metals and alloys, two synergistically affecting each other processes take place simultaneously – a change in the structure and properties of the base metal and the appearance of oxide films on the surface. During the oxidation, the following iron oxides are formed: FeO (wustite), Fe<sub>3</sub>O<sub>4</sub> (magnetite) and Fe<sub>2</sub>O<sub>3</sub> (hematite). Oxides are arranged in layers according to the decrease in oxygen content from the outer to the inner layer (Figure 1).



Figure 1. Simplified scheme of oxide films on the surface of carbon steel during heating in an oxygen-containing environment

Wustite (FeO) has face-centered cubic (FCC) and stable at temperatures above 570 °C. Below this temperature, it decomposes. The largest amount of wustite formed in the temperature interval 700–900 °C. Wustite is the softest and loosest layer of the oxide film. Magnetite (Fe<sub>3</sub>O<sub>4</sub>) has a cubic

crystal lattice, highly abrasiveness and is insoluble in acids. Hematite (Fe<sub>2</sub>O<sub>3</sub>) has the highest abrasiveness and practically does not dissolve in acids [1]. With a metal hardness according to Vickers of 140 units, the hardness of FeO is 270–350, Fe<sub>3</sub>O<sub>4</sub> 420–500, Fe<sub>2</sub>O<sub>3</sub> 1030 units.

In mechanical engineering, the study of processes that occur during heating and cooling of steel, as an alloy of iron with carbon and other elements, is typically limited to the study and optimization of the base metal's structure. The composition, thickness, and properties of oxide films are dealt with either by specialists in hot rolling [2–5], where scale (a high-temperature oxide film on the surface of steel) is defective, or by specialists in processing scale and restoring iron from it<sup>1</sup> [6–8].

Therefore, the purpose of this research is to determine the effect of temperature loading on the hardness and magnetic properties of hypoeutectoid steel, which depend both on the processes occurring in the base metal and on the properties of surface oxide films.

## 1. Methods and materials

The work is experimental. For the experiment, cylindrical samples made of carbon steel 25 (carbon content from 0.25–0.33%) were used. The critical points of the researched steel are  $Ac_1 = 735$  °C,

<sup>&</sup>lt;sup>1</sup> Degai AS, Zuev MV, Zasukhin AL, Karmanov OB, Mikurova MI, Orekhov OE, Gusev RV. *Method of preparation of oiled scale for processing*. RU2279491C2. 10.07.2006. (In Russ.)

 $Ac_3 = 835$  °C. To study the coercive force ( $H_C$ , A/m), a verified and certified structuroscope (coercimeter) KIM-2M was used. Figure 2 shows a sample and nozzles for measuring coercive force. For hardness, the Rockwell method B scale (HRB, dimension arbitrary units) was chosen, implemented in a stationary hardness tester TK-14-250. The samples were heated in a laboratory furnace with a PM-16M-V thermostat. The experiment was carried out as follows: the samples were placed in a furnace heated to a given temperature, held for 15 minutes, and cooled in calm air. The temperature ranged from 200 to 1000 °C. In each sample, before heating and after cooling, the hardness and coercive force were measured several times along the length of the sample, and then the values were averaged. Before heating, for all samples – the average value of hardness is 89 units of the Rockwell B scale, the average value of the coercive force is 1023 A/m.



Figure 2. Sample and nozzles for measuring coercive force

#### 2. Results and discussion

On Figure 3, numbers from 1 to 9 show the heating temperatures of the samples under study, plotted on the steel corner of the Fe–Fe<sub>3</sub>C diagram. Samples 1–6 were heated to temperatures below the critical point Ac<sub>1</sub>, sample 7 – between Ac<sub>1</sub> and Ac<sub>3</sub>, samples 8 and 9 were completely austenitized during heating. All samples, except the first, were heated above the Curie point of cementite (210 °C). Research the behavior of the coercive force during thermal cycling near the Curie point of cementite (210 °C) is the subject of works [9–13]. However, the study of the complex influence of processes in the base metal and in the zone of formation of oxide films on engineering characteristics – hardness and magnetic properties during single heating has not been carried out.

Figures 4 and 5 are show graphical dependences of the experimental results. Numbers 1–9 correspond to the temperatures of the experiment in Figure 3.



Figure 3. The steel angle of the Fe–Fe3C diagram, with points (1–9) corresponding to experiment temperatures



**Figure 4.** Dependence of the change in hardness after heating and cooling on the heating temperature: *a* – average hardness before heating; *b* – after heating and cooling



Figure 5. Dependence of the change in the coercive force after heating and cooling on the heating temperature: *a* – the average value of the coercive force before heating; *b* – after heating and cooling

Figure 6 shows the temper colors of the samples after heating and cooling. Sample 0 is one of the samples at the same experimental series, not participating in the experiment. Obviously, the visual assessment of temperature by temper colors is subjective, when the temperature threshold of 500 °C is exceeded (in this work), the visually determined dependence between the temperature and the color

of the sample surface after exposure to temperature disappears.

In this experiment, both the coercive force and hardness were determined taking into account the properties of the base metal and surface oxide films. The depth of magnetization of the device used and the depth of penetration of the indenter when measuring hardness are significantly greater than the thickness of the total oxide films, therefore, the measurement result is a complex value depending on the properties of the base metal and oxides. Description of the processes occurring in steel during experimental studies is given in Table.



Figure 6. Temper colors of samples after heating and cooling

Description of the occurring processes	in the research samples	during heating and cooling
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Tomporatura interval	Popotion of bordnoop and opprojug force	Steel shanges
Temperature Interval	Reaction of naroness and coercive force	Steer changes
200–300 °C	Slight drop from unloaded temperature state	Residual stresses decrease in the base metal, a thin single-layer oxide film appears on the surface
300-400 °C	Hardness reduction continues. Coercive force decreases slightly	Deformed crystal lattice begins to align in the base metal; hematite predominates in the structure of sur- face oxides (sample 3, Figure 6)
400–500 °C	Increased hardness and coercive force	There is a similarity of incomplete annealing in the struc- ture of the base metal, but the thickness of a particu- larly hard layer (hematite) increases on the surface
500–600 °C 600–700 °C	Hardness drop. Avalanche-like drop in coercive force due to the appearance of non-ferromagnetic elements in the structure of surface oxide films	Incomplete annealing of the base metal occurs, loose wustite predominates in the structure of the surface layers (appeared at a temperature of 570 °C)
700–800 °C	Hardness drop. Growth of the coercive force due to the predominance of the wustite ferromagnetic component in the oxide structure, the phenome- non of scale detachment from the base metal	The transition during heating to the GSF region of the Fe–Fe3C diagram (Figure 2), an increase in the thickness of oxide films on the surface
800–900 °C	Hardness increase. Sharp drop in coercive force	Complete austenization of the base metal, hardening takes place in it due to a process of similar normaliza- tion, partial removal of oxides from the surface occurs
900–1000 °C	Sharp drop in hardness and coercive force	There is an overheating of the base metal, uncontrolled growth of austenite grains, self-removal of scale from the surface (Figure 7)



Figure 7. High temperature scale on the sample surface

## Conclusion

When applying a temperature load and subsequent cooling in carbon steel, two synergistically influencing processes occur simultaneously – a change in the structure and properties of the base metal and the appearance of oxide films on the surface. Hardness and coercive force (the most structurally sensitive magnetic characteristic of a ferromagnet) are not correlated with each other at all temperature intervals of heating. It is obvious that the visual assessment of temperature by temper colors is subjective, when the temperature threshold of 500 °C is exceeded (for the research steel in given modes); the visually determined dependence between the temperature and the color of the sample surface after temperature exposure disappears. Therefore, recommendations for determining the temperature of the fire effect on metal structures by the temper colors of steel structures (for example, after a fire) cannot be recognized as sufficiently reliable.

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