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Hands-on Experience of THERMO FITNESS TESTING Device Use for Thermoelectric Evaluation of Metallic Materials

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Abstract

The article describes experience of practical application of the differential thermoelectric tester "THERMO FITNESS TESTING". Description of the sensor design and results of differential thermoelectromotive force measurement for a large group of metals widely used in Russia are given. Usage of the "THERMO FITNESS TESTING" device to test the quality of R6M5 steel heat treatment is described. Dependence of thermoelectromotive force on the heating temperature which can be used for practical purposes was obtained. Usage the "THERMO FITNESS TESTING" to measure the thickness of a cemented (carburized) layer of 12KH2N4A steel is considered. Dependence of the thermoelectromotive force on the thickness of the cementation layer was also obtained.

Keywords: ThermoEMF, differential sensor, electrode, Seebeck coefficient, nondestructive testing

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Опыт применения прибора термоэлектрического контроля металлов и сплавов «THERMO FITNESS TESTING»

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Приведена информация о применении прибора термоэлектрического контроля «THERMO FITNESS TESTING» для контроля металлов и сплавов, которые широко распространены в России. Особенностью прибора является дифференциальный датчик, в конструкции которого используется два нагреваемых электрода. Датчик снабжён системой нагрева и стабилизации температуры горячих электродов. Нагреваемые электроды имеют одинаковую температуру, что позволило получить высокую повторяемость измерения термоЭДС. Описан также пример использования прибора «THERMO FITNESS TESTING» для контроля качества термообработки стали Р6М5. Получена зависимость термоЭДС от температуры нагрева под закалку, которую можно использовать для практических целей. Рассмотрен еще один пример использования прибора «THERMO FITNESS TESTING» для контроля толщины цементованного слоя стали 12Х2Н4А. Также получена зависимость термоЭДС от толщины слоя цементации.

Ключевые слова: ТермоЭДС, дифференциальный датчик, электрод, коэффициент Зеебека, неразрушающий контроль

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Introduction

After discovery of the Seebeck effect, the thermoelectric method for the testing of metal and alloy products became widely used [1–6]. Thermo-electromotive force (thermoEMF) occurs in a closed circuit of two conductors, where junctions are at different temperatures. This condition is provided by the hot and cold electrodes of the thermoelectric detector, which provide two junctions with different temperatures in contact with the tested product. The thermoEMF value depends on the temperature difference between the hot and cold junction, therefore it must remain unchanged during the measurement process. Thermoelectric testing devices utilize measurement systems of absolute [7–19] and differential thermopower [20–23]. The differential thermoEMF measurement system is preferred due to the higher noise immunity, sensitivity, and stability of the instrument readings. This is due to the same temperature regime at the contact points of the hot electrodes. The simplest testing system based on differential thermopower is shown in Figure 1.

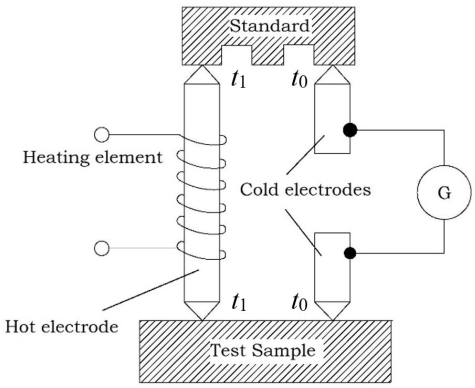


Figure 1 – The simplest differential thermoelectric measurement system

The circuit consists of a sensor, a standard reference, a test specimen, and a galvanometer. The sensor contains a hot electrode with a heater and two cold electrodes. One electrode has a higher temperature than the other. The electrode of the higher temperature (hot electrode) is at temperature t_1. The other electrode of the lower temperature (cold electrode) is at temperature t_0, which is usually at ambient temperature. A galvanometer connected to two cold electrodes forms a closed circuit together with a reference, hot electrode, and a test specimen. The differential thermoEMF depends on the temperature difference and the Seebeck coefficients of both samples:

$$E_{dif} = \Delta T(a_x - a_{ref}), \tag{1}$$

a_{ref} is the Seebeck coefficient of the standard, a_x is the Seebeck coefficient of the test specimen; $\Delta T = t_1 - t_0$.

Errors in measuring differential thermoEMF are caused by two factors:

1. Incorrect determination of temperature difference ΔT at measurement points due to imperfection of temperature sensors and their calibration [24].

2. Mismatch of points between which the temperature difference ΔT and the differential thermoEMF E_{dif} are measured.

In addition, the differential sensor shown in Figure 1 is inconvenient for practical use, because it has three independent electrodes: one hot and two cold, and its use in mobile devices is difficult. In addition to the above, it is necessary to provide four contacts: the first is the contact of the hot electrode with the standard, the second is the contact of the hot electrode with the test specimen, the third is the contact of the first cold electrode with the standard and the fourth is the contact of the second cold electrode with the test specimen.

Design features of the developed device

Authors have developed and implemented an original sensor design which is easy to use and is an integral part of the developed differential thermoelectric tester.

The main technical characteristics of the thermoelectric tester are given in Table 1. It should be noted that thermoelectric testing devices are indicator-type devices.

The sensor design and sensor's position on the tested and reference samples is shown in Figure 2*a*, whereas Figure 2*b* shows a photograph of the sensor.

It uses two heated electrodes E_1 and E_2 with a common heater. Heated electrodes are made of the same metal or alloy. This sensor design is very convenient for practical use. When measuring differential thermopower, it is necessary to ensure that electrical and thermal contacts of the hot electrodes with the reference and the test specimen are established. Moreover, one hot electrode is only in contact with the reference, and the other is only in contact with the test specimen, while a common heater for the heated electrodes ensures the same temperature at both contact points. In the sensor housing, the electrodes are glued together into a common structure, and an insulating fiberglass gasket is placed between them. A heating element is placed in the resulting round hole inside the electrodes along with a temperature sensor just like the ones installed in temperature-controlled soldering stations. The electrodes are placed in the sensor housing. The resulting thermoEMF is the sum of the EMF of the contact pairs included in the measuring circuit of the sensor:

$$E_{dif} = E_1 \pm E_2 - E_3 + E_4 - E_5, \quad (2)$$

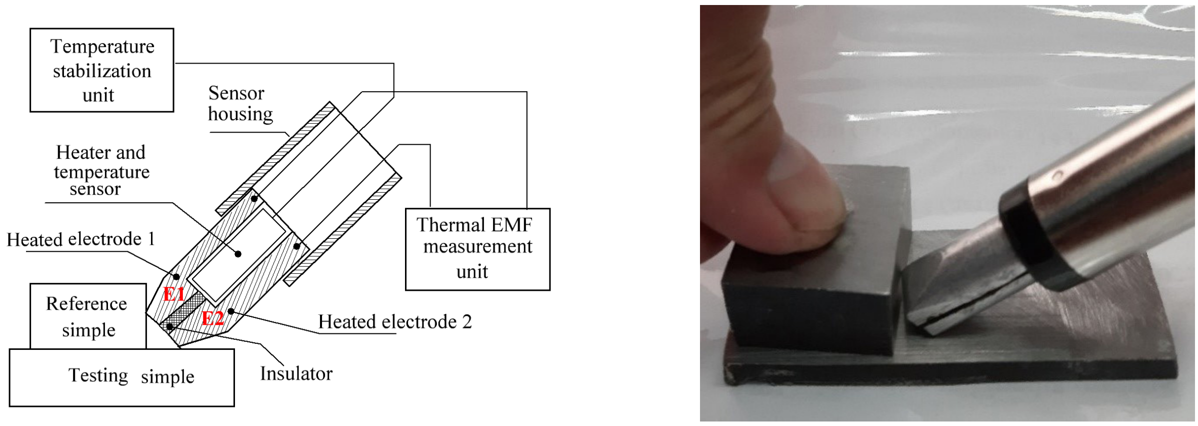
where E_1 is the EMF of the metal pair the first hot electrode- reference; E_2 is the EMF of the metal pair reference-test specimen; E_3 is the EMF of the metal pair the second hot electrode-test specimen; E_4 is the EMF of the metal pair the first hot electrode-copper connecting conductor; E_5 is EMF of the pair of metals: the second hot electrode is a copper connecting conductor. Since the heated electrodes are made of the same alloy, we can write:

$$E_4 = E_5. \quad (3)$$

Table 1

Technical characteristics of the developed differential thermoelectric tester

Parameter	Unit of Measurements	Magnitude
Supply voltage	Volt	220
Sensor temperature	Degree Celsius	130±3
Temperature accuracy	%	±5
Threshold voltage during sorting	μV	0.01…0.4
ThermoEMF measurement range	μV	0…±9.99
Device readiness time	minutes	15
Turning on the "Accepted" signal	The indicator is green	EMF is less than threshold voltage
Turning on the "Not accepted" signal	The indicator is red	EMF is greater than threshold voltage



a *b*

Figure 2 – Differential sensor: *a* – schematic representation of the sensor; *b* – photograph of the sensor

Taking into account expression (3), formula (2) will take the form:

$$E_{dif} = E_1 \pm E_2 - E_3. \quad (4)$$

The reference can be of any shape, because thermoEMF does not depend on the size and shape of the sample. Technologically, it is easiest to produce a reference metal or alloy in the form of a parallelepiped.

Value of thermopower is influenced by various factors: structural condition, heat treatment, coating, chemical composition, etc., therefore, the standard reference and the test specimen should have only one distinctive feature. For example, if the chemical composition is inspected, then the other distinguishing features should be the same (heat treatment mode, coating, structural condition, etc.).

It should be noted that not all groups of steel can be inspected. According to Russian GOST standards carbon steels are divided into 3 main groups (A, B, C), where Group A steel is usually used for parts that are not hot in the manufacturing process, Group B steel is used for parts that undergo hot forming (stamping, forging) or heat treatment, and Group C steel is typically used for welded structures. For example, Group A steels, cannot be inspected due to the different chemical composition of the same steel grade. The steels of groups B and C can be inspected, because they have the same chemical composition. The preparation of samples for inspection includes cleaning from rust, oil and dirt. Moreover, cleaning cannot be carried out with a file or a similar tool to exclude the ingress of tool particles into the contact area of the electrodes, which will entail a change in thermopower.

If the reference and the test specimen are the same in chemical composition and structural state, then the thermopower E_1 and E_3 will be the same, but opposite in sign, and E_2 will be zero. If there are deviations in the chemical composition or structural state of the tested specimen from the standard, the thermoEMF E_1 and E_3 will be different. E_2 will not be equal to zero, however, considering the fact that the contact temperature corresponds to the ambient temperature, its value will be several orders of magnitude lower compared to E_1 and E_3. The compliance criterion can be selected by the user by changing the value of the "threshold". In most cases, this value corresponds to 0.1 μV and is associated with the tolerance of the chemical composition of the same grade of steel produced at different times or by different manufacturers.

The proposed differential sensor design makes it possible to reduce overall dimensions and increase ease of use, especially in a portable version powered by a battery for operation at sites where there is no power supply network (oil pipelines, cold warehouses for storing supplied metal, etc.).

Application

For acceptance inspection and testing of compliance with the Russian GOST standard of supplied products and sorting of finished products by steel and alloy grades, thermoEMF measurements of the most widely used steel grades in Russia were carried out using the developed differential thermoelectric tester for metals and alloys.

Results of differential thermoEMF measurements are given in Table 2, where columns list different steel grades used in the manufacture of the standard reference, and rows indicate test specimens.

Table 2

Values of differential thermoEMF of some of the widely used steel grades, denoted in Russian Cyrillic alphabet between parentheses

Steel grade	9KHS (9ХС)	R6M5 (Р6М5)	3KH3M3F (3Х3М3Ф)	4KH4VMF (ДИ22)	KH12MF (Х12МФ)	6KHV2S (6ХВ2С)	U8A (У8А)	KH12F1 (Х12Ф1)	KHVG (ХВГ)
9KHS (9ХС)	0 ±0.05	−0.95 ±0.05	− 1.35 ±0.05	−0.90 ±0.05	−1.25 ±0.05	−0.45 ±0.05	−0.60 ±0.05	−1.30 ±0.05	−0.80 ±0.05
R6M5 (Р6М5)		0 ±0.05	−0.30 ±0.05	+0.15 ±0.05	−0.25 ±0.03	+0.55 ±0.05	+0.30 ±0.05	−0.30 ±0.05	+0.25 ±0.05
3KH3M3F (3Х3М3Ф)			0 ±0.05	+0.40 ±0.05	+0.10 ±0.05	+0.70 ±0.05	+0.75 ±0.05	+0.12 ±0.05	+0.50 ±0.05
4KH4VMF (ДИ22)				0± 0.05	−0.35 ±0.05	+0.45 ±0.05	+0.40 ±0.05	−0.30 ±0.05	+0.20 ±0.05
KH12MF (Х12МФ)					0± 0.05	+0.70 ±0.03	+0.60 ±0.05	−0.10 ±0.05	+0.45 ±0.05
6KHV2S (6ХВ2С)						0 ±0.03	−0.15 ±0.03	−0.85 ±0.05	−0.25 ±0.5
U8A (У8А)							0 ±0.03	−0.70 ±0.05	−0.10 ±0.05
KH12F1 (Х12Ф1)								0 ±0.05	+0.55 ±0.05

Continuation of Table 2

Steel grade	20KH (20Х)	SHKH15 (ШХ15)	KH12M (Х12М)	KH12F1 (Х12Ф1)	30 (Ст.30)	45 (Ст.45)	U8 (У8)	U12 (У12)
20KH (20Х)	0 ±0.05	0.50 ±0.05	0.90 ±0.05	1.90 ±0.05	0.50 ±0.05	0.60 ±0.05	0.75 ±5	0.80 ±0.05
SHKH15 (ШХ15)		0 ±0.05	1.25 ±0.05	2.00 ±0.05	0.12 ±0.05	0.20 ±0.05	0.50 ±0.05	0.55 ±0.05
KH12M (Х12М)			0 ±0.03	1.00 ±0.05	1.35 ±0.05	1.45 ±0.05	1.60 ±0.05	1.65 ±0.05
KH12F1 (Х12Ф1)				0 ±0.03	2.05 ±0.05	2.15 ±0.05	2.40 ±0.05	2.45 0±0.05
30 (Ст.30)					0 ±0.05	−0.10 ±0.05	0.10 ±0.05	0.15 ±0.05
45 (Ст.45)						0 ±0.05	0.20 ±0.05	0.25 ±0.05
U8 (У8)							0 ±0.05	0.05 ±0.05

From Table 2 it can be noted that some pairs have very close thermoEMF values. For example, KH12F1 steel and KH12MF steel, as well as KHVG steel and U8A steel. In Table 2, the pairs of steel 30 and steel 45, as well as steel U8 and steel U12, have similar thermoEMF values. Therefore, errors are possible during verification of supplies and sorting of finished products of these pairs of steels. Other steel grades have a greater difference in thermoEMF and there are no problems with their sorting.

The developed thermoelectric testing device for metals and alloys was used to inspect the quality of heat treatment of R6M5 steel, of which 10 samples were used to study the dependence of thermopower on the heating temperature. The samples were heated in a CHO 24/1250 furnace with a temperature range of 1150–1250 °C. The step of temperature change was 10 degrees. Quenching was carried out by cooling in oil. A sample of heat-treated steel of the same grade, heated to 1225 °C, was used as a reference. The resulted dependence of measured differential thermoEMF on temperature is shown in Figure 3. The confidence interval is 7 %.

Before inspecting another steel grade, it is necessary to conduct preliminary studies on the dependence of thermoEMF on the heating temperature. In addition, it should be considered that when heated, a decarbonized layer appears on the sample surface, which has a strong effect on the value of thermoEMF, therefore it must be removed before measurement.

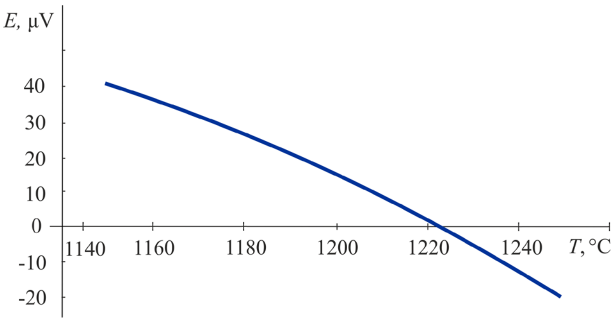


Figure 3 – Dependence of differential thermoEMF on heating temperature for hardening of R6M5 steel

The main controlling parameter of chemical and thermal treatment is the depth of the saturated layer. To inspect the depth of the saturated layer, the metallographic method is traditionally used, which is a direct inspection method that is capable of determining the depth of the cemented or nitrided layer with high accuracy. Nonetheless, this method of inspection is considered destructive because it requires the preparation of a metallographic specimen and, in addition, the testing procedure is lengthy. To assess the possibility of using the developed differential thermoelectric tester to inspect the depth of the cemented layer, the dependences of the differential thermoEMF on the depth of the layer on the sample were plotted. To study the dependence of thermopower on the thickness of the cemented layer, samples of 12X2H4A steel were prepared, subjected to cementation, quenching and tempering. The change in the thickness of the cementation layer was achieved by grinding the surface of the samples. The size of the sanded layer was 0.2 mm each time. At the same time, the thickness of the cemented layer was inspected metallographically. After each grinding, the differential thermoEMF was measured. The standard reference was made of the same grade of steel but was not cemented.

The differential thermoEMF along the depth of the ground layer was determined at each step using 5 measurements. The time of one measurement is no more than 3 s. The obtained dependence (Figure 4) can be used as a calibration dependence to determine the depth of the cemented steel layer.

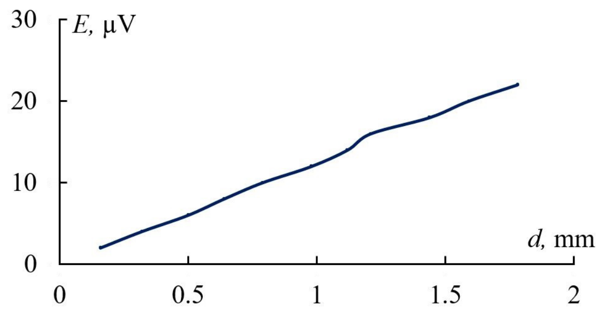


Figure 4 – Dependence of the differential thermoEMF on the thickness of the cemented layer

The confidence interval for the experimental dependence presented in Figure 4 is 6.5 %.

As can be seen from Figure 4, the change in differential thermoEMF from the thickness of the cemented layer is almost linear.

To inspect the thickness of the cemented or nitrided layer on another steel grade, it is necessary to first obtain its dependence of the thermoEMF on the thickness of the cemented layer and use it for calibration.

Conclusion

The thermoelectric tester developed by the authors has found application in various industries for incoming inspection of supplied steel, such as quality control of heat treatment, determining the presence of a decarburized layer and its depth, and determining the thickness of the steel carburization layer. All commercially produced portable thermoelectric testing devices do not implement the differential sensor due to its bulky and inconvenient design. Hence, their main drawback is the change in thermoEMF when the hot electrode is cooled due to heat transfer to the cold sample under test. The authors proposed an original design of a differential sensor that ensures the same temperature regime of hot electrodes, as well as ease of operation with such a sensor. A simultaneous change in the temperature of two hot junctions leads to a change in the absolute emf of each hot junction, but the difference remains unchanged.

A linear dependence of thermoEMF on the heating temperature for hardening R6M5 steel has been established, which can be used to assess the quality of heat treatment. Moreover, with an increase in the heating temperature for hardening from 1150 to 1250 °C, the thermoEMF decreases from 40 μV to −20 μV. The authors also established a linear dependence of thermoEMF on the thickness of the cemented layer of steel 12X2H4A. With an increase in the thickness of the cemented layer from 0.02 to 1.8 mm, the thermoEMF increases from 2 to 22 μV. This relationship can be used to evaluate the quality of cementation.

To sort widely used steel grades, during the practical use of the device, differential thermoEMF was measured for 15 steel grades. Most of them have large differences in thermoEMF and can be accurately determined. However, minor differences (0.05±0.05 μV) were revealed for such pairs as: KH12F1 – KH12MF, KHVG – U8A, Steel 30 – Steel 45 and U8 – U12, which makes it difficult to sort these steel grades.

One of the possible ways to solve this problem, according to the authors, may be a multi-temperature regime. If the thermoEMF of the marked pairs of metals varies in temperature (different slope of the characteristics or their nonlinearity), it is possible to measure the thermoEMF at several hot junction temperatures and process the obtained data. This field of research is a key focus for the authors.

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