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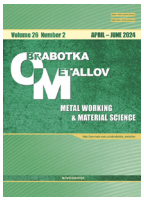

Oxidation temperatures of WC-Co cemented tungsten carbides

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ABSTRACT

Introduction. Products containing *WC-Co* cemented tungsten carbides are commonly used in various industries. It is often operates at elevated temperatures, at which, as noted in the literature, tungsten carbides are susceptible to severe oxidation in air. However, no sufficiently accurate values of oxidation temperatures and dependence of these temperatures and the oxidation rate of tungsten carbides on the cobalt content with its wide variation have been established. ***The subject*** of the study is the oxidation process of *WC-Co* cemented tungsten carbides. **The purpose** of the work is to obtain the oxidation temperatures of *WC-Co* cemented tungsten carbides with different cobalt content by weight in the range of 3–20 %. **Methods.** The dynamics of oxidation was studied in air. Specimens of the same length were heated to a temperature of 850 °C and cooled at the same rate in the furnace of a push-rod dilatometer *Netzsch 402 PC* while its expansion was simultaneously recorded. The oxidation rate of the specimens was determined by the difference in its length before heating and after cooling. The values of oxidation temperatures were obtained by mathematical analysis of relationships of the expansion on temperature. **Results and discussion.** Experimental dependences of expansion of *WC-Co* cemented tungsten carbides on temperature in the range from 20 to 850 °C, and for *WC-8Co* – up to 1,150 °C, were obtained. The oxidation rate of *WC-Co* cemented tungsten carbides increased linearly with increasing concentration of tungsten carbides (decreased with increasing cobalt content). During heating, two characteristic temperatures were identified: the onset of oxidation (631±4 °C) and the transition to active oxidation (804±11 °C). The established temperatures were the same for different ratios of tungsten carbides and cobalt. The results can be used when choosing temperature conditions for products made from *WC-Co* cemented tungsten carbides.

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Introduction

Tool *WC-Co* cemented tungsten carbides are widely used in various industries. Products containing these alloys are often used in a range of elevated temperatures, which determines the relevance of studying its performance under these conditions. The properties of most materials change significantly as temperature increases, and an increase in chemical activity plays a special role, in particular, when interacting with atmospheric oxygen. The fact of high-temperature oxidation of *WC-Co* cemented tungsten carbides is confirmed by a number of researchers.

The oxidation of *WC-Co* cemented tungsten carbides is weak up to 600–650 °C [1–3], only cobalt is slightly oxidized [4]. In the temperature range of 650–800 °C, there is a sharp increase in the oxidation rate [3, 5–8], which is confirmed by the curves obtained by the thermogravimetric analysis (*TGA*). At temperatures above 800 °C, intense oxidation begins. The longer the thermal exposure time (even at constant temperature), the greater the thickness of the oxide layer, which is confirmed by all researchers.

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This leads to a significant increase in the size of the specimen [3, 9, 10], which is due to the porosity of oxides formation, which favors unhindered access of oxygen. Oxidation occurs not only during a long-term isothermal process [11, 12], but also during a short-term exposure to laser radiation [13].

It is noted in [14] that the oxidation rate increases with increasing temperature; however, the study [15] revealed a local inversion in the dynamics of oxidation in the temperature range of 528–630 °C. The oxidation rate also increases with the increasing heating rate [1, 5], which is confirmed by a shift in the curves of the dependence of mass on temperature. However, this may be due to the specifics of the experiment.

It is noted in [6] that in the temperature range up to 650 °C a linear oxidation law is observed (dependence of the increase in mass on temperature), and above 800 °C it is quasi-parabolic. In other words, the oxidation rate increases linearly with increasing temperature above 800 °C.

Increased oxygen [1, 12] and air flow rates accelerate oxidation [1], but at high flow rates this effect weakens due to cooling of the specimen.

Recent studies have shown that *WC-Co* cemented tungsten carbides undergo several stages of oxidation when heated [16]: extremely weak oxidation of cobalt and carbides (up to 600 °C), the onset of oxidation (above 600 °C) of cobalt (*CoO*, Co_3O_4), and simultaneous oxidation of cobalt and carbides (above 700 °C) with the formation of tungsten oxides (WO_2, WO_3) and double oxides of tungsten and cobalt ($CoWO_4$) [1, 7, 16]. A similar pattern is observed not only for sintered materials, but also for a mixture of powders [17]. A number of studies have noted that cemented carbides with a higher cobalt content oxidize more slowly than those with a lower content [1, 18, 19].

In [7], the authors discovered that the hardness of *WC-10Co* with particularly fine grains drops significantly during oxidation. However, the *Vickers* hardness of a surface cleaned of oxides after cooling may either not change at all or even increase [20]. During oxidation, the bending strength of cemented carbides decreases [12, 21, 22], which is largely due to the degradation of the surface layer. This was indirectly confirmed in [23], where the influence of oxidation on the development of surface cracks was noted.

Coatings have a protective function [3, 24] and prevent oxidation of the cemented carbide base. However, when heated (due to thermal stresses caused by differences in the thermal coefficient of linear expansion), cracks appear in the coatings, allowing oxygen access to the base. In addition, coatings themselves are also oxidized [25].

Not only specially prepared specimens with a ground surface were used, but also commercially produced cutting inserts [26], including those with coatings [24] were used to study the oxidation process of cemented carbides. The results presented coincide with the results obtained on special specimens.

Oxidation can be used to purposefully reduce the strength of a cemented carbide in order to improve its machinability [27]. In the work [8] it is even proposed to use oxidation to recycle worn multifaceted cutting tool inserts. However, when using cemented carbides as cutting tool materials, its high-temperature oxidation can play an important [28–30] or decisive role in the wear of cutting tools [26, 31, 32].

Having studied the literature, we can see that the above experiments mainly used isothermal heating with a large step of temperature change (500, 600 °C, etc.). The accuracy of the determined oxidation onset temperatures was not sufficient, and the influence of cobalt content on the value of these temperatures and on the dynamics of oxidation with wide variations in the composition of *WC-Co* cemented tungsten carbides was not established.

The quantitative study of oxidative processes can be performed in various ways: by weight changes of the specimen – thermogravimetric analysis (*TGA*); by temperature difference – differential thermal analysis (*DTA*); by changes in heat flux – differential scanning calorimetry (*DSC*); by changes in the parameters of thermal radiation – infrared spectroscopy [15]; by changes in the properties of reflected radiation – optical or scanning electron microscopes (*SEM*). However, these methods do not always provide trustworthy information about the oxidation rate, and its accuracy is limited by the nature of the experiment. For instance, in thermogravimetric analysis, due to the absorption of gases and the appearance of volatile oxides in addition to solid oxides, the change in the mass of the specimen can be ambiguous.

Since the density of the substance changes during chemical reactions, the dimensions of the specimen also change. This makes it possible to quantify oxide layers directly by comparing the dimensions of the specimen before and after heating. However, this requires interrupting the heating process and removing the specimen from the furnace.

Dilatometers designed to determine the thermal coefficient of linear expansion (*TCLE*) of a material can also be used to measure the thickness of oxide formations directly during heating [33, 34].

This paper is a continuation of the work of the authors [35] on dilatometry of *WC-Co* cemented tungsten carbides and ***its purpose*** is to determine the temperatures at which the oxidation of these cemented carbides with different cobalt content begins, as well as to establish the relationship of cobalt content with the oxidation temperatures and the dynamics of oxide formation. Achieving this purpose requires studying the dynamics of the *WC-Co* oxide growth with a wide variation in cobalt content by heating to high temperatures in a laboratory furnace and by dilatometry.

Methods

Small specimens were heated in a laboratory furnace in air atmosphere to qualitatively assess the dynamics of oxidation. For this purpose, six specimens with cobalt content *Co* = 3, 6, 8, 10, 15, 20 wt. % were prepared. These specimens were made from commercially produced carbide cutting tool inserts and cylindrical blanks for milling cutters. The composition of *WC-Co* cemented tungsten carbides corresponded to the *VK* (*WC-Co*) group of the Russian Standard [36]. The *Fisher* grain size of tungsten carbide powders was 4.0–9.0 μm (according to the manufacturer). The specimens' sizes ranged from 3.5 to 5 mm. In the furnace, the specimens' were placed on a corundum substrate and heated to 900 °C at a rate of 7 °C/min in air, after which, without holding at the maximum temperature, naturally cooled to 20 °C without removing them from the furnace.

The most common and reliable push-rod dilatometer *Netzsch 402 PC* was used in this work to quantify the dynamics of oxidation. The dilatometer's push rod and specimen holder are made of alumina ceramics (Al_2O_3). Tests were carried out in air. The specimen was heated and cooled at a rate of 7°C/min. The contact force of pressing the push rod against the specimen was 0.35 N. To measure the temperature of the specimen, an *S*-type thermocouple was used, located in close proximity to it. Nitrogen was supplied to its housing at a flow rate of 30 ml/min to protect the displacement sensor.

Specimens for dilatometry with a 5×5 mm square cross section were made from the same blanks as the specimens for heating in the furnace. The nominal length of the specimens corresponded to the length of the reference specimen used to calibrate the dilatometer and was 25±0,001 mm. To remove the surface layer obtained during sintering of the workpieces, the ends of the specimens in contact with the holder and push rod were polished with a diamond grinding wheel. The initial length of each specimen was determined at room temperature using a micrometer with a 1 μm scale division in three dimensions.

Prior to the experiment, the dilatometer was calibrated at a constant heating rate of 7 °C/min to 1,200 °C using a standard Al_2O_3 reference specimen. The test specimens were heated at the same rate to 850 °C. One specimen with cobalt content *Co* = 8 % was heated to 1,150 °C. Cooling to room temperature was carried out at the same rate as heating. Thus, the time of thermal exposure of the specimen was the same for all specimens heated to 850 °C. Simultaneous recording of temperature and specimen expansion was conducted continuously one time per second.

The data obtained during dilatometry were approximated by cubic splines. The characteristic temperatures of oxidation were determined from the inflection points of both lines (heating and cooling) of the thermal expansion curves by finding the extrema of the first and second derivatives. The resulting (after cooling) thickness of the oxide layers was determined using the thermal expansion curves that have a characteristic hysteresis to determine the average oxidation rate of each specimen.

The experimental results were processed using standard tools in *MS Excel* by fetching subroutines specially written in *MATLAB* with the built-in functions for working with splines using the *Spreadsheet Link* plugin.

Results and discussion

Table 1 presents the results of heating the specimens of *WC-Co* cemented tungsten carbides with different cobalt content by weight in a laboratory furnace to 900 °C. The top row of the table shows the initial appearance of the specimens, and the bottom row – its appearance after heating and cooling. As we can see from the photographs, all *WC-Co* cemented tungsten carbides are highly oxidized. The oxide layers are of considerable thickness and in some cases peel off from the base. The cross sections of these layers correspond to the shape of the surfaces on which they are formed. All specimens changed color. None of the specimens had traces of interaction with the corundum substrate. The experimental results confirm that *WC-Co* cemented tungsten carbides highly oxidize with the material structure being destroyed when heated to temperatures above 800 °C. These results are consistent with the data obtained by other researchers for other cobalt contents [3, 5–8].

Table 1

Results of heating *WC-Co* specimens with different cobalt content in air in a laboratory furnace

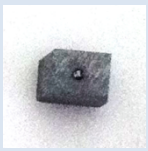
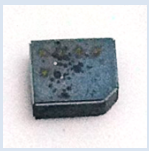
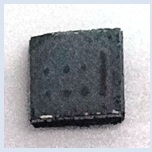
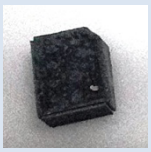
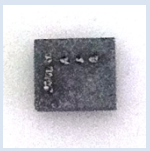
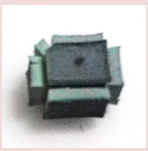
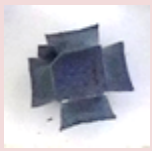
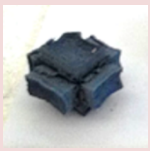
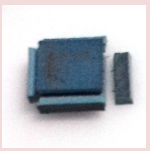
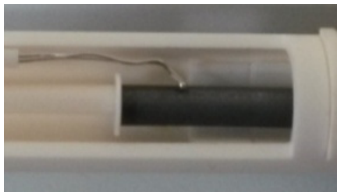
Co, %	3	6	8	10	15	20
20 °C						
900 °C						

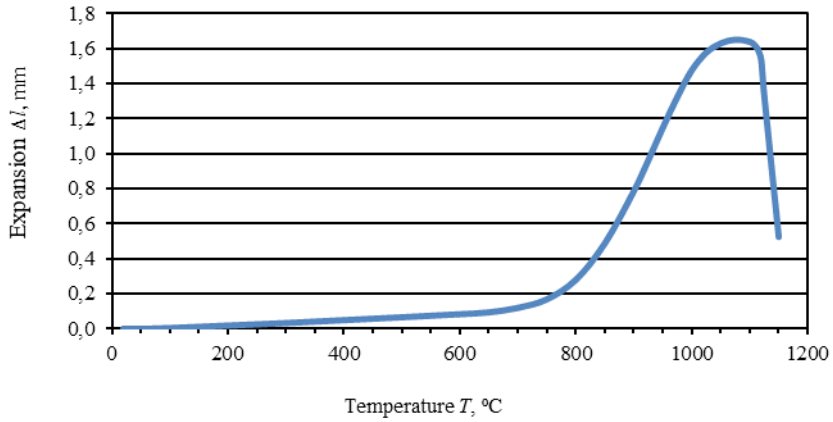
Fig. 1 shows the results of heating a specimen of a *WC-Co* cemented tungsten carbides with a cobalt content of *Co* = 8 % in a dilatometer to a temperature of 1,150 °C. The appearance of the specimen before heating is shown in Fig. 1, *a* and after completion of the experiment in Fig. 1, *b*. The last photo demonstrates that the specimen has significantly deteriorated and lost its shape. The surfaces of the specimen holder and push rod were covered with a blue coating. Fig. 1, *c* shows the relationship between the specimen expansion Δl and temperature *T*. The curve shows three characteristic temperature ranges: slight expansion up to about 650 °C, a sharp increase in size from 650 °C to approximately 950 °C, and destruction of the specimen at a higher temperature. In all these ranges there are sections of the curve close to the linear law. In the first range, changes are caused almost exclusively by the thermal expansion of the specimen with a slightly increasing *TCLE* [35]. In the second range, thermal deformation is accompanied by the growth of the oxide layer, which begins to dominate. If in the range of 20–650 °C the specimen increased by only 0.096 mm, in the range of 650–1050 °C the specimen increased by 1.534 mm. In the third temperature range, the increase in specimen size slows down and then the specimen shortens, which can be explained by loss of shape and destruction of the specimen.

Fig. 2 shows relationships between the expansion and temperature for *WC-Co* specimens with different cobalt content, obtained by heating to 850 °C and subsequent cooling to room temperature. All the curves are similar and have a characteristic hysteresis, indicating permanent elongation due to the presence of oxide layers. The section of the curves corresponding to cooling looks like a mirror image of the heating section. The first two characteristic temperature ranges of slight expansion and a sharp increase in specimen size are observed.



a

b



c

Fig. 1. View of the specimen (*Co* = 8 %) in the dilatometer before heating (*a*) and after heating (*b*); graph of the dependence of the specimen expansion on temperature (*c*)

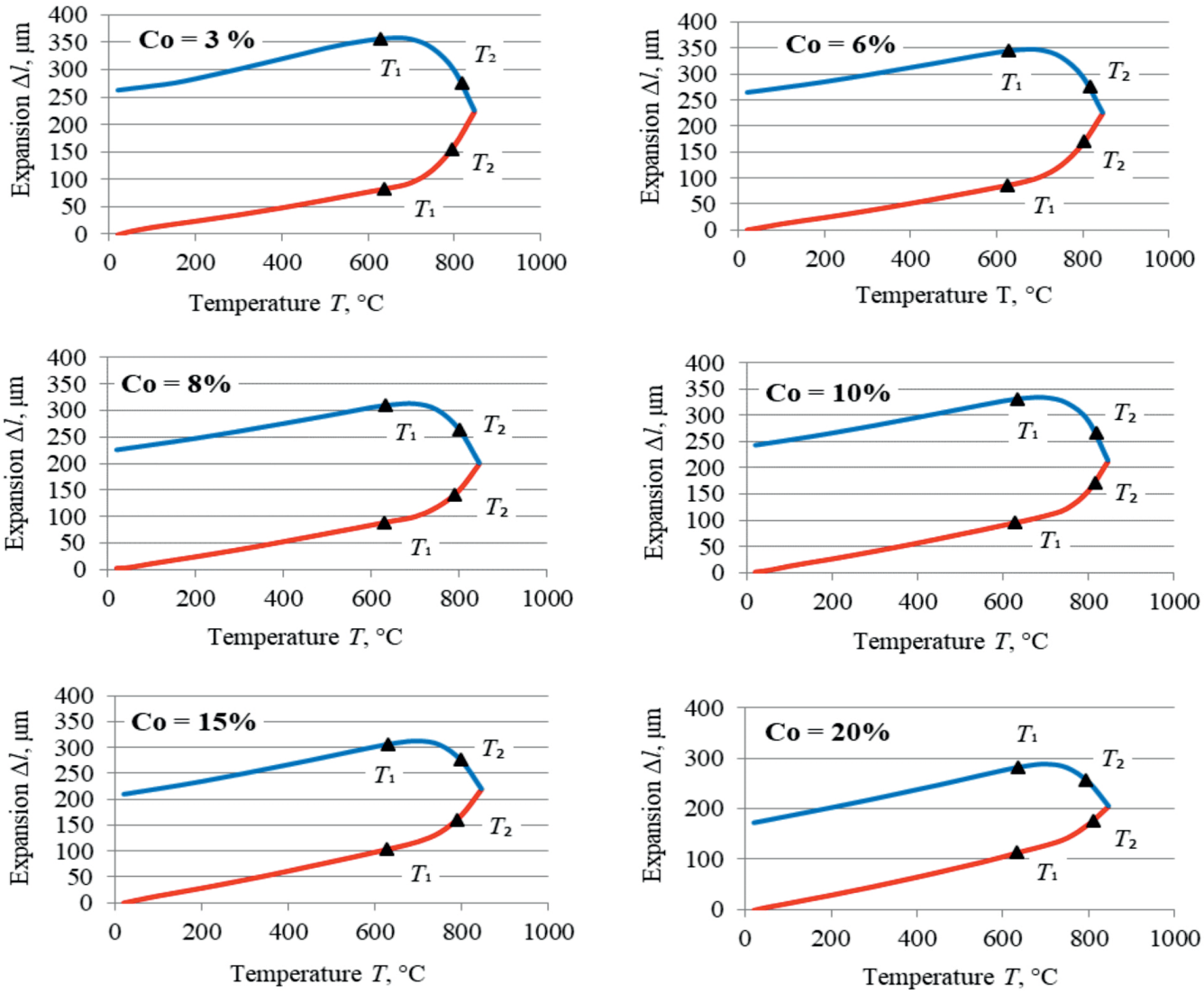


Fig. 2. Dependences of the expansion on temperature for *WC-Co* specimens with different cobalt content

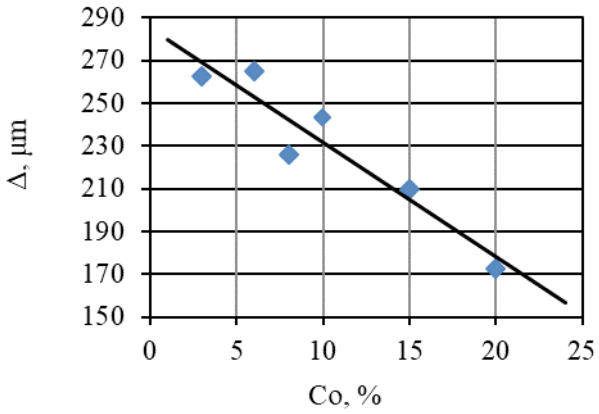


Fig. 3. Dependences of the specimen change in length **Δ** (after heating and cooling) on cobalt content

As a result of processing the data shown in Fig. 2, a dependence of the specimen change in length **Δ** (after heating and cooling) on cobalt content was obtained (Fig. 3) by linear approximation (coefficient of determination R^2 = 0.897). Since the testing time is the same for all specimens, the residual elongation corresponds to the average oxidation rate. Thus, the average oxidation rate is inversely proportional to the cobalt content and increases with increasing content of tungsten carbides. This dependence is close to the linear law in the considered range of changes in the cobalt content in the alloy. Data on the effect of cobalt content on the oxidation rate for the tungsten carbides considered are consistent with the results of previous studies obtained for: *WC-6Co* and *WC-12Co* [1]; *WC-6Co*, *WC-10Co* and *WC-18Co* [12]; *WC-15Co* and *WC-25Co* [18] under isothermal conditions.

Figs. 4 and 5 show curves of the first and second derivatives (for heating and cooling, respectively, see Fig. 2) of the dependences of expansion Δl on temperature T for *WC-Co* specimens with different cobalt content for the range of 550–850 °C, which is of greatest interest. The graphs generally confirm the inverse dependence of the average oxidation rate on cobalt content based on residual elongation. The second derivatives have two characteristic inflections: around 630 °C (T_1) and 800 °C (T_2). Before T_1, the first derivative has a horizontal section, and the values of the second derivative are close to zero. Closer to T_1, both derivatives begin to increase significantly, which indicates the emergence of new chemical processes (mainly, carbide oxidation with WO_3 formation and cobalt oxidation) that affect the change in size. This point corresponds to the onset of oxidation of the cemented carbides. Around T_2, an extremum at the maximum value of the second derivative of the heating line, as well as an extremum of the second derivative of the cooling line are observed. The obtained temperature values at T_2 correlate with the temperature values of characteristic points on the curves of the heat flux changes obtained in previous studies [3, 5–7] using the differential scanning calorimetry (*DSC*) method. T_2 corresponds to the transition to the active oxidation of the carbide, after which the ratio of $CoWO_4$ in the total weight of the oxide layer probably begins to increase. The beginning of the graphs' slowdown after T_2 can be explained by the higher density of $CoWO_4$ compared to WO_3. Domination of $CoWO_4$ at high temperatures is confirmed by spectral analysis of the oxide layer after heating *WC-Co* [3].

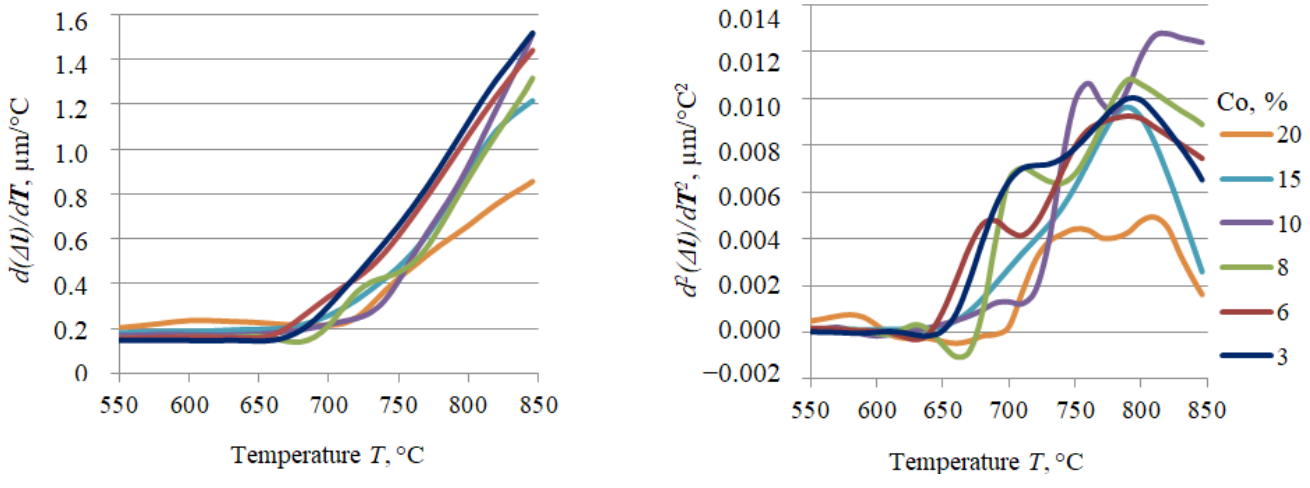
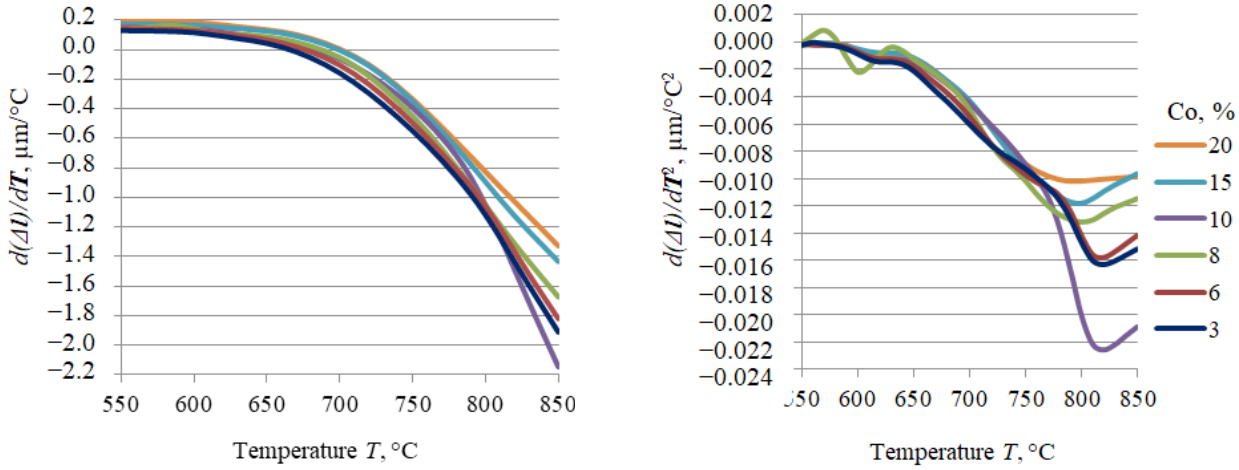


Fig. 4. Graphs of the first (*a*) and second (*b*) derivatives of the relationship of expansion Δl on temperature T for *WC-Co* specimens with different cobalt content obtained during heating



a *b*

Fig. 5. Graphs of the first (*a*) and second (*b*) derivatives of the relationship of expansion **Δ*l*** on temperature ***T*** for *WC-Co* specimens with different cobalt content obtained during cooling

An intermediate extremum in the lines of the second derivative for heating may result from the errors caused by the mechanism of the dilatometer and is not observed in cooling.

Table 2 summarizes the values of characteristic points T_1 and T_2 for all *WC-Co* cemented tungsten carbides obtained during heating (Fig. 4) and cooling (Fig. 5). Fig. 6 shows graphs of the dependence of T_1 and T_2 on cobalt content, where the black lines show the average values and the red lines show the root-mean-square (*RMS*) deviations from these values.

Table 2

Temperatures T_1 and T_2 during heating (↑) and cooling (↓) of *WC-Co* with different cobalt content

Temperature		Co, %						Mean value
		3	6	8	10	15	20	
T_1	↑	638.8	626.2	630.6	628.6	628.4	633.7	631.1
	↓	628.1	627.7	631.6	634.6	631.2	634.1	
T_2	↑	793.5	803.9	791.2	815.1	789.6	809.7	804.1
	↓	818.5	816.8	801.4	818.5	797.5	793.8	

Analysis of the results shows that the values of T_1 and T_2 during heating and cooling for different cobalt content are very similar. For T_1, the maximum deviations from the average value were 1.2 %, and for T_2 the maximum deviations from the average value were 1.8 %; the *RMS* deviations were 0.6 % and 1.4 %, respectively. In absolute terms, the *RMS* deviations for T_1 were ±4 °C, and for T_2 the standard deviations were ±11 °C. At the same time, changes in actual temperature values depending on cobalt content is insignificant and random, which is apparently due to experimental errors. Thus, we can conclude that the quantitative composition of *WC-Co* cemented tungsten carbides has virtually no effect on the temperatures of the onset of oxidation and the transition to active oxidation.

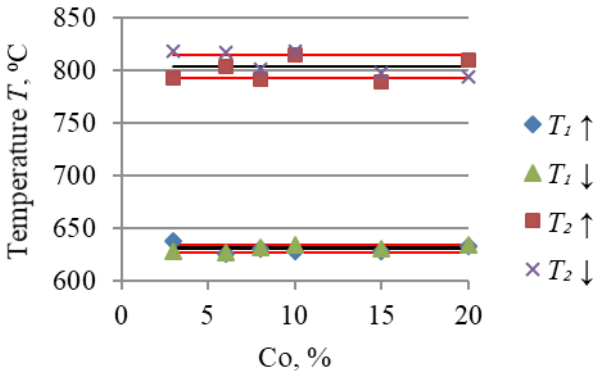


Fig. 6. Dependences of characteristic temperatures T_1 and T_2 on cobalt content, obtained during heating (↑) and cooling (↓)

This is apparently due to the properties of the tungsten carbides, which do not significantly depend on the concentration of the cobalt phase.

The resulting temperatures of the onset of oxidation (631±4 °C) and the transition to active oxidation (804±11 °C) are in good agreement with the ranges of characteristic temperatures of oxidation obtained by other researchers using methods other than the one used here.

Conclusions

1. Oxidation of *WC-Co* cemented tungsten carbides with a wide variation in cobalt content (Co = 3–20 wt. %) heated to elevated temperatures was studied.

2. After heating in a laboratory furnace to 900 °C, strong oxidation of these tungsten carbides was observed, with the material structure being destroyed.

3. Dilatometric studies resulted in obtaining experimental graphs of the expansion of *WC-Co* cemented tungsten carbides specimens at temperatures up to 850°C. The graphs have a characteristic hysteresis, indicating permanent elongation due to the presence of oxide layers. The two characteristic temperature ranges of slight expansion and a sharp increase in specimen size are observed.

4. For a specimen with a cobalt content of 8 %, an additional experiment was carried out with heating to 1,150 °C; as a result, it was completely destroyed.

5. The average rate of oxidation of *WC-Co* cemented tungsten carbides increases with increasing content of tungsten carbides (decreases with increasing cobalt content) and this relationship is linear.

6. Two characteristic temperatures were identified: the onset of oxidation (631±4 °C) and the transition to active oxidation (804±11 °C). These temperatures are the same for different ratios of tungsten carbides and cobalt.

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Conflicts of Interest

The authors declare no conflict of interest.