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Fundamentals of High Temperature Corrosion

Oxidation Under Thermal Shock Conditions

GOAL

The goal of this exercise is to become familiar with research methodology and evaluation of scaling-resistant metallic materials oxidized in cyclic temperature change conditions. Additionally, the effect of a protective coating on the scaling-resistance of selected materials oxidized in thermal shock conditions will be studied.

INTRODUCTION

The consequence of subjecting metallic materials simultaneously to an oxidizing atmosphere and a high temperature is formation of single or multiphase oxide layers (called scales) on the surface of these materials. When a pure metal is oxidized, the grown scale is built of the appropriate oxide of the given metal. However, when a multicomponent alloy is oxidized, the scale can be built of a mixture of constituent metal oxides or of the alloy component with the highest affinity towards oxygen in given conditions (so-called selective oxidation). The build and adherence of the scale to the substrate depends on whether it will exhibit good protective properties during further oxidation. Due to rapid temperature changes during heating and cooling the material as it works, stresses will be generated between the scale and substrate as a result of, among other things, different thermal expansion coefficients of the metallic core and the oxide layer, which is, in fact, a ceramic material. A sufficiently high concentration of these stresses, as a consequence, leads to the scale, which serves as a protective layer, cracking and spalling off the substrate. This will, in turn, accelerate metallic substrate degradation and limit the life-time of construction elements exposed to the effects of not only high temperature and an aggressive gas environment, but also rapid temperature change, the rate of which can even be up to several hundred (and more) degrees Celsius per minute.

Even if a metallic material, after losing part of its protective scale, is able to reproduce this layer by means of further oxidation of the component that forms the protective oxide, continuous spallation is associated with accelerated consumption of that element. As a further consequence, this leads to such changes in the chemical composition of a scaling-resistant alloy that further reproduction of the protective layer will be impossible. Chemical composition changes can also result in changes in the phase composition/microstructure, which can lead to deterioration of e.g. the alloy mechanical properties. At this moment, very rapid metal degradation will take place making it unsafe to continue using the given construction element.

In order to determine the scaling-resistance limit and lifetime of the materials destined for operation at high temperatures, experimental studies, sometimes very time-consuming, are necessary. These studies enable relatively easy determination of the degree of scale-substrate adhesion. The idea behind these studies is to heat materials to a high temperature many times and keep them at that temperature for a certain period of time. Next, the samples are very rapidly cooled to room temperature and then weighed in order to determine mass changes caused by formation or loss of the protective part of the scale as a function of number of cycles. Fig. 1 presents an interpretation of the possible mass changes per unit area as a function of the number of thermal shocks: from very good, where scale spallation either doesn't take place or is very minor, to very poor, where most of the grown oxide spalls off during each oxidation cycle.

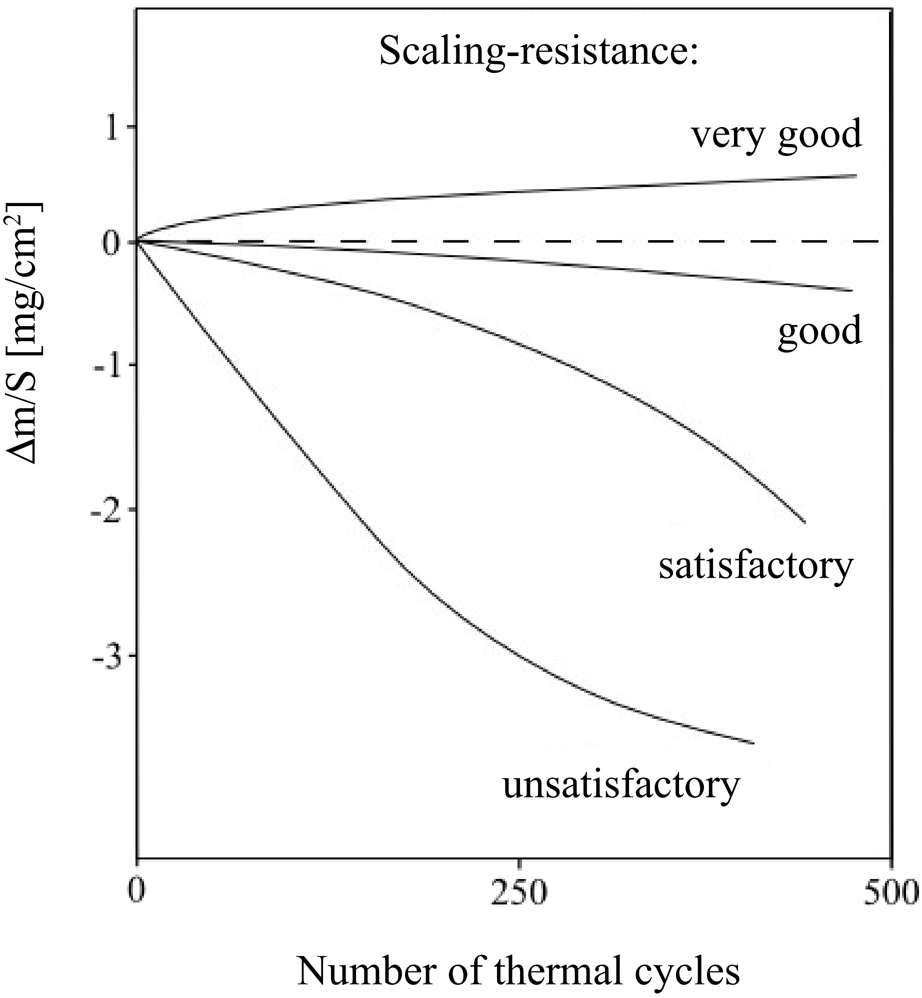

Fig. 1. Cyclic oxidation courses for materials of different scaling-resistance.

Selection of oxidation parameters: temperature, cycle length, atmosphere and number of repetitions is dependent on the needs of the given studies and can be chosen in wide ranges.

The test method of corrosion rate measurement in cyclic temperature change conditions provides valuable services when it comes to evaluating the scaling-resistance of materials destined for operation in several branches of industry. However, due to the complexity of the processes that can take place in real conditions, the method is often modified in various ways, as a result of which individual methods of evaluating scaling-resistance can sometimes significantly differ from one another. This is the main reason large repeated differences in evaluations pertaining to the durability of studied materials.

PERFORMING THE EXERCISE

In the framework of this exercise, a cyclic oxidation test is to be carried out at 850°C on three materials chosen by the laboratory instructor (e.g. Cu, Cu covered by a Ni layer, scaling-resistant steel). One full cycle consists of heating the materials at the high temperature (10 min.), cooling down to room temperature (5 min.) and weighing the samples. After that the cycle is repeated.

The three previously selected samples must be cleaned with sandpaper until mirror shine is achieved, their surface areas must be measured, they must be purified in ethyl alcohol, then completely dried and weighed on an analytical scale. After preparing the samples in the previously-mentioned way, insert the materials into the apparatus for cyclic oxidation. Before that, the furnace should be heated to the desired temperature. Set the durations for a heating cyclic and cooling (see above). Every time after a heating cyclic is finished, wait until the samples have completely cooled down and then weigh them on the above-mentioned analytical scale. Once again, insert the samples into the apparatus and oxidize them according to the previously provided instructions.

NOTE: When removing the samples for weighing be especially careful due to the possibility of contact with apparatus elements that still exhibit a high temperature and keep the samples away for your eyes. As a result of oxide scale spallation, you could damage your eyes (protective glasses required).

ELABORATION OF RESULTS

Present the obtained results on a cumulative plot that illustrates mass changes of all the samples per unit area as a function of a number of oxidation cycles. On the basis of the plot determine which of the materials exhibits the best and worst scaling-resistance. Present observations and conclusions about oxide scale formation on the different materials that underwent cyclic oxidation and pay attention to the effects of the Ni coating on the Cu substrate compared to the uncoated Cu sample.

Literature

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