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Corrosion of metals and alloys — Electrochemical potentiokinetic reactivation measurement using the double loop method (based on Čihal's method)

*Corrosion des métaux et alliages — Mesurage de la réactivation
électrochimique potentiocinétique par la méthode de la double boucle
(dérivée de la méthode de Čihal)*



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 12732 was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

Corrosion of metals and alloys — Electrochemical potentiokinetic reactivation measurement using the double loop method (based on Čihal's method)

WARNING — This International Standard may involve hazardous materials, operations and equipment. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices, and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies the method for measuring the degree of sensitization (DOS) in stainless steel and nickel-based alloys using the Double Loop Electrochemical Potentiokinetic Reactivation (DL-EPR) test (based on Čihal's method).

The method may be used for the quantitative assessment of deleterious thermal effects resulting in the formation of alloy-element-depleted zones at grain boundaries or in the matrix. However, attention should be paid when testing heat-affected weld zones, due to possible non-uniform distribution of sensitized zones along the fusion lines.

The results of the test can be used as an index to identify the potential susceptibility of stainless steel and nickel-based alloys to intergranular corrosion, pitting corrosion, and intergranular-stress corrosion cracking, but prediction of these corrosion modes depends on complementary specific testing.

This International Standard describes the general methodology and, in Annex C, gives examples of suitable test exposure conditions for specific alloys.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 8044:1999, *Corrosion of metals and alloys — Basic terms and definitions*

ISO 643:2003, *Steels — Micrographic determination of the apparent grain size*

3 Terms and definitions

For the purposes of this document, the terms and definitions in ISO 8044 and the following apply.

3.1

integrated charge

Q

charge measured during passivation (Q_p) and reactivation (Q_r), given by the time integral of current below the passivation and reactivation peak of the curve

4 Principle

Heat treatment (including welding) of corrosion-resistant alloys can lead to formation of particles, such as chromium carbide in the case of 304 SS or σ -phase (FeCrMo) in duplex stainless steels. This will lead to local depletion of alloying elements, unless replenished by matrix diffusion, the extent of which will be temperature dependent. This process is commonly referred to as sensitization, because depleted zones have an intrinsic lower resistance to localized corrosion and, where appropriate, to stress corrosion cracking. The extent to which these damage mechanisms develop and propagate will depend on the extent of depletion and the density of depleted zones. There was a need for a simple laboratory test to rapidly identify potentially deleterious thermal effects on stainless steels and nickel-based alloys. The EPR test was developed for that purpose. Although two methods have been used in laboratory testing, the single loop and the double loop, the former, which involves polarization scanning from the passive to the active state, has the disadvantage that the method can be sensitive to surface finish. The EPR test may also be applied to in-field testing, provided some adaptation be made to the cell and assembly.

The double loop version of this method is preferred. Here, the specimen is immersed in an acid solution such that it is in the active state under freely corroding conditions, but then anodically polarized into the passive domain. As surface features are dissolved during initial immersion under active corrosion conditions, the likelihood of surface preparation having an impact diminishes. From the passive state, the specimen is polarized at a controlled scan rate in the cathodic direction. A schematic illustration is shown in Figure 1. In the absence of active depleted zones, the passive film can become unstable as the potential becomes less positive and can start to dissolve (e.g. by reductive dissolution). However, the rate of dissolution is small and, with the sweep rate employed, the anodic current is not able to rise substantially, so that only a modest anodic peak current is measured. The process of anodic dissolution during the cathodic scan is referred to as reactivation. Occasionally, the current may go transiently cathodic on lowering the potential, as the passive current density may be less than the cathodic current at the potentials of relevance.

When a depleted zone is present, the passive film is locally less protective and is more easily reduced. Hence, active dissolution of the depleted zones will occur more readily whilst adjacent material still retains some passive film, albeit a gradually thinning passive film. Thus significant active dissolution occurs. The reason for the gradual rise in current is probably a reflection of the spread in activity associated with a spread of the extent of depletion and corresponding variations in the passive film properties (some sections reduced at higher potentials and some at lower potentials). Gradually, as the activity of all the sites build up, this begins to be counteracted by the decrease in potential reducing the current of the active regions because of Tafel behaviour; thus a reactivation peak is observed. The peak in the current density, and the charge passed associated with that peak, depend on the extent of alloy depletion.

The ratio of the reactivation peak to the activation peak, or the reactivation charge to the activation charge, when compared with the values for the solution-annealed specimen, gives an index of sensitization. However, for sensitized grain boundaries, these values have to be normalized to the grain size. At the end of the test, the specimen is examined to confirm the nature of the localized corrosion process.