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論文内容要旨

Chapter 1 Introduction

The exposure of steel at different temperatures in manufacturing industries leads to the formation of oxide scales. The evaluation of oxide layer properties is important to researchers and manufacturers because it has a great influence in different fields under material production and management. Different properties must be taken in account for better understanding the influences of the oxide layer. There are several methods for determining the properties of the oxide layer. The properties of oxides formed on large metallic surfaces at different temperatures are difficult to measure nondestructively in-service. In the research work, a spring-loaded macroscopic direct current (DC) four-point probe potential drop (PD) technique is used to nondestructively evaluate the properties of the oxide layer deposited on a large metallic surface. The DC four-point probe PD technique has advantages over other techniques for evaluating the properties of the oxide layer. When the metallic surface is covered with an oxide layer, PD measured by four-point probe technique contains the contributions of dual channels current flow. These dual channels are made of a top thin oxide layer and a bottom bulk base material. The effect of top oxide layer in the measurement of PD cannot be ignored. The technique through the present method is capable of receiving simultaneously the effects of the oxide layer and the base material. The method is a new and unique attempt in the field of nondestructive testing and evaluation (NDT&E).

Chapter 2 Checking Surface Contamination and Determination of Electrical Resistivity of Oxide Scale by DC Potential Drop Technique

This chapter describes the nature of potential drops (PDs) on carbon steel (SS400) and stainless steel (SUS304) samples. The spring-loaded macroscopic DC four-point probe sensor is used for the measurements of PD on both of the oxidized and the oxide scale free surfaces. In the study the oxidized test surface is considered as two layers of different resistivities materials as shown in Fig.2-1. The test sample consists of two layers; the top layer, i.e., oxide scale having resistivity ρ_1 and thickness t , and the bottom layer, i.e., base material having resistivity ρ_2 , and large thickness compared to the top layer. The effect of two layers on the PDs is illuminated by an electrical image method. A physical model of a two-layer test sample based on the experimental conditions through electrical image method is developed.

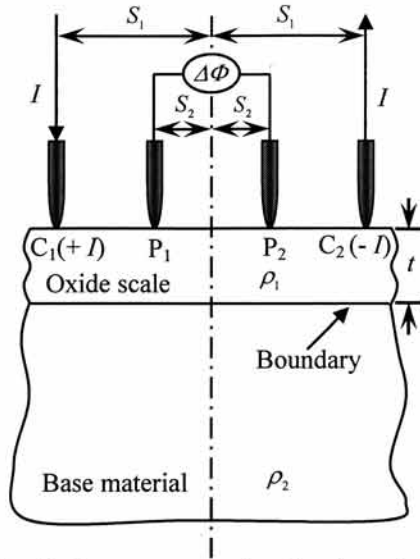


Fig.2-1 Probes arrangement on two-layer test surface.

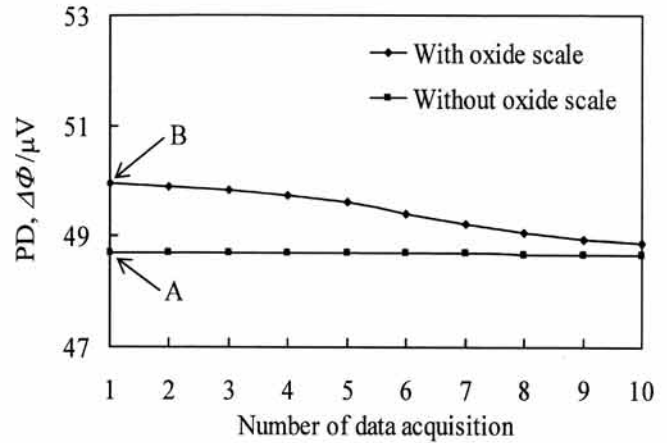


Fig.2-10 Comparison of nature of PDs on carbon steel for one contact in 15 s.

The parameters $2S_1$ and $2S_2$ are distances of current probes and PD measuring probes, respectively, as shown in Fig.2-1. The current I is injected into the material via the outer probes. The potentials Φ_1 and Φ_2 at the measuring probes P_1 and P_2 are due to the current source and drain probes C_1 and C_2 . The potential drop between two measuring probes $\Delta\Phi$ [$=(\Phi_1 - \Phi_2)$] is given by Eq.(2-14). On the other hand, the test sample behaves as a single layer when it is free from oxide scale. The potential drop for this case can be expressed by Eq.(2-15). These equations are given as

$$\Delta\Phi = \frac{2\rho_1 I}{\pi} \times \frac{S_2}{S_1^2 - S_2^2} \left(1 + 2 \sum_{m=1}^{\infty} k^m \right) \quad (2-14) \quad \text{and} \quad \Delta\Phi = \frac{2\rho_2 I}{\pi} \times \frac{S_2}{S_1^2 - S_2^2} \quad (2-15)$$

, where k [$=(\rho_2 - \rho_1)/(\rho_2 + \rho_1)$] is reflection coefficient. The experimental results show the remarkable nature of PDs on oxidized test surfaces with probe contact time. The nature of PDs on the oxidized and the oxide scale free surfaces are compared for the same experimental conditions and it is easy to compare the contaminated surface with the oxide scale free surface and to decide whether the surface is oxidized or clean. The PDs decrease on the oxidized test surface and remain constant on the oxide scale free surface with probe contact time as shown in Fig.2-10. The PDs at positions A and B in Fig.2-10 can be used to determine the resistivities of base material and oxide scale by using Eqs.(2-15) and (2-14), respectively. Electrical resistivity of the oxide scale is successfully determined by the DCPD technique on the basis of the two layers of different resistivities model. In an attempt to prove the validity and verify the accuracy of the proposed method, measurements of PD on the oxidized surface are performed with various probes spacing having a pitch of 10 mm. Then, the electrical resistivity is also determined for various probe spacing and all of the results are shown to be very close to one another.

Chapter 3 Evaluation of Electrical Resistivity of Oxide Scale Formed under Different Conditions and Observation of the Basis of Decreasing Nature of Potential Drop on Oxidized Surface

In this chapter, the nature of DCPD on carbon steel (SS400) having different surface conditions are investigated. Three samples from the same type of carbon steel are prepared under three different conditions, namely, short and long period exposures in atmosphere and low temperature heat treatment of short exposure sample. The decreasing nature of PD of oxidized surface is varied from one to another sample. The PD decreasing nature with probe contact time for the

oxidized test samples depends on the thickness and/or the mechanical properties of the oxide scale. The PDs of oxidized surface decrease due to the deformation of the oxide layer under the tip of the current probes. As the oxidized test sample is of two layers with the thin oxide layer and the thick base material, a small part of supplied current will flow through the oxide layer. Therefore, the total supplied current I can be divided into I_1 and I_2 for the oxide layer and the base material, respectively, as shown in Fig.3-2. As observed in Fig.2-10, the potential drops on oxidized test surface decreased with probe contact time. This indicates that the current densities in the layer under the measuring probes were decreased with probe contact time. The tip of the current probe moves towards the surface of more conductive base material due to deformation of the oxide layer under the probe tip as shown in Fig.3-2. It is worth understanding that the DCPD technique through spring-loaded macroscopic four-point probe is able to distinguish the oxidized surface from the oxide scale free surface. Electrical resistivities of oxide scale in all of the three cases are successfully evaluated through the developed two layers model. The almost unique value of evaluated electrical resistivity of oxide scale in all of the cases indicates that the oxide materials are similar in type in respect to electrical resistivity.

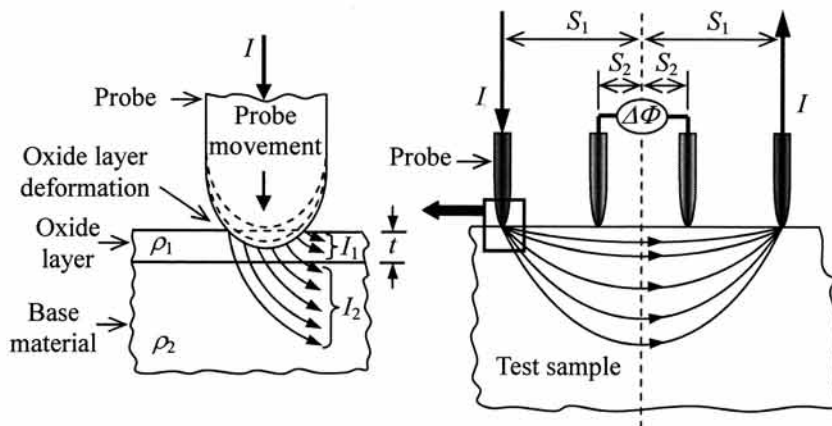


Fig.3-2 Schematic diagram for the phenomenon of decreasing nature of potential drop.

Chapter 4 Nondestructive Evaluation of the Thickness of an Oxide Layer Deposited on a Large Surface Using the DC Potential Drop Technique

The DCPD measurement system is used to measure the PD on oxidized test surfaces for different spring force values. In this study, the force of the spring attached to the current probes is reduced by inserting spacers with different thicknesses under the supporting legs of the sensor block. The insertion of the spacer under the legs reduces the compressed length of the spring. It also indicates that an insertion of a thick spacer under the legs decreases the force of the spring attached with current probes. The PD measurement is first performed on a machined sample exposed to the atmosphere for approximately six months. The PD for the oxidized surface decreased until the tip of the probe reached the interface between the oxide layer and the base material. As shown in Fig.4-6, the probe contact time required to achieve a constant PD increased with increasing spacer thickness. Then, the thickness of the oxide layer deposited on the surface of the sample is increased by heat treatment. The PD is also measured on the samples subjected to heat treatment. The experimental results establish a relationship between spring force and the time required for the current probe to penetrate the oxide layer as shown in Fig.4-7. Then, this study proposes a procedure for evaluating the thickness of the oxide layer. The proposed method is successfully used to determine the thickness of the oxide layer deposited on a large surface. To demonstrate the validity and accuracy of the proposed method, the obtained thickness

is compared with the thickness measured directly using a field emission scanning electron microscopy (FE-SEM).

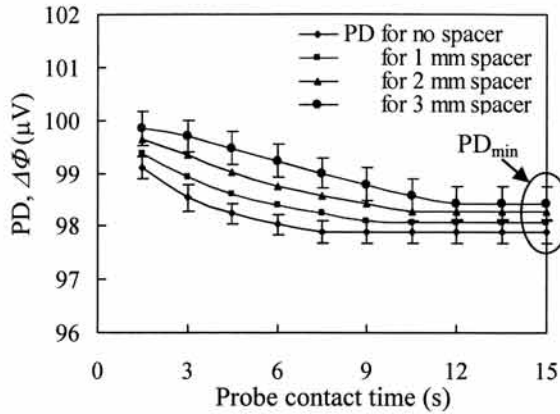


Fig.4-6 Nature of PD for the sample exposed to atmosphere.

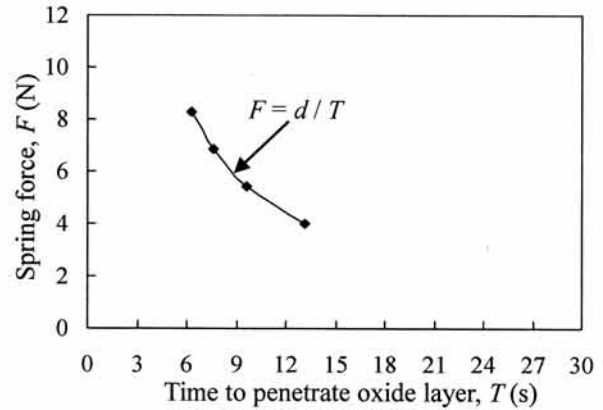


Fig.4-7 Effect of spring force on the time to penetrate the oxide layer for the sample exposed to atmosphere.

Chapter 5 The Effect of the Oxide Layer on Effective Heat Transfer Coefficient for Oxidized Metal

It is important to know the effects of the oxidized surface on heat transfer for cooling steel materials in the production process. In this chapter, the heat transfer phenomenon from oxidized metallic surfaces to surrounding medium was clearly discussed. The nondestructively measured thickness of the oxide layer deposited on a large metallic surface is successfully used to evaluate the effective heat transfer coefficient. The effect of the oxide layer on the effective heat transfer coefficient of cooling system in materials production processes is clearly presented. The effective heat transfer coefficient decreased with an increase in the thickness of the oxide layer. For the case of the free convection heat transfer, there is no significant influence of oxidized surface with a smaller thickness of the oxide layer on the effective heat transfer coefficient. On the other hand, for the case of the forced convection heat transfer, both of the smaller and the larger thicknesses of the oxide layer have a significant influence on the effective heat transfer coefficient.

Chapter 6 Conclusions

The DCPD technique is capable of receiving the influences of inhomogeneity of the oxidized sample in the measurements of PD. A physical model of two layers test sample based on electrical image method was developed for measurements of PD. The PDs decrease on the oxidized test surface and remain constant on the oxide scale free surface with probe contact time. The decreasing nature of PDs indicates that the current densities in the layer under the measuring probes were decreased with probe contact time due to movement of the tip of the current probe towards the surface of more conductive base material through deformation of the oxide layer under the probe tip. The electrical resistivity of the oxide layer deposited on a large surface was successfully determined using the developed two layers model. The developed method is suitable for different conditions of the sample. Furthermore, a simple and attractive procedure to nondestructively evaluate the thickness of the oxide layer was established. The thickness of the oxide layer deposited on a large surface was successfully determined using DC four-point probe PD technique. Finally, the effective heat transfer coefficient for oxidized metal is shown to be successfully determined using nondestructively evaluated thickness of the oxide layer. For the case of the forced convection heat transfer, both of the larger and the smaller thicknesses of the oxide layer have a significant influence on the effective heat transfer coefficient.

論文審査結果の要旨

機械・構造用金属材料表面に形成される酸化層は、これらの腐食に対して保護膜としての役割りを果たすと同時に、熱伝達、摩擦等の表面での諸現象に大きな影響を与える因子である。そのため酸化層の特性を評価することは従来から重要視されてきた。しかしながら非破壊的な評価については、酸化層の厚みが小さいこと等の困難さにより、研究が遅れている状況にあった。著者は、直流電位差法により、酸化層の電気抵抗率ならびに厚みを評価できる手法を見出し、酸化層非破壊評価の基礎を構築した。本論文は、これらの研究成果をまとめたものであり、全編6章からなる。

第1章は序論であり、本研究の背景、目的および構成を述べている。

第2章では、はじめに母材と酸化層からなる2層材を対象にした直流電位差法による鏡像法に基づく電流問題の理論式を詳細に検討している。次に、酸化層を有する炭素鋼表面上で電流を入出力し、電位差を計測したとき、計測電位差が時間経過に伴わずかに減少し、やがて一定値を示すようになる現象を見出し、計測開始時の電位差と上記理論式より、酸化層の電気抵抗率をその厚さに関係なしに評価する手法を提案し、ステンレス鋼に対する電位差計測結果も踏まえて、その有効性を検証している。これは、酸化層特性の非破壊評価手法の基礎を与える重要な成果である。

第3章では、種々の条件下で形成された炭素鋼表面上の酸化層を対象にした実験より、時間経過に伴う計測電位差の減少が、電流入出力端子先端が酸化層を貫通する方向に移動することによることを示し、かつ計測開始時の電位差の使用が酸化層の電気抵抗率評価に妥当であることを再確認している。これらは第4章の議論を展開するに先立ち基盤となる成果である。

第4章では、電流入出力端子を被検査材表面に押付ける荷重が計測電位差の減少現象に及ぼす影響を明らかにし、これを踏まえて、酸化層の厚みを評価する手法を提案し、断面観察による酸化層の厚みの直接計測と比較して、検証している。これは、酸化層の厚みの直流電位差法による非破壊評価手法のはじめの提案であり、貴重な成果である。

第5章では、酸化層を介した熱伝達に関する議論を記述している。これは、第4章の成果の応用を指向した重要な一適用例の指摘である。

第6章は結論である。

以上要するに本論文は、直流電位差法により酸化層の電気抵抗率ならびに厚みを評価できる手法を提案、検証し、酸化層非破壊評価の基礎を開拓したものであり、ナノメカニクスおよび機械工学の発展に寄与するところが少なくない。

よって、本論文は博士(工学)の学位論文として合格と認める。