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Response of Oxide Scales to Energetic Particle Impact: Initial Results and Evaluation of Experimental Technique

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RESPONSE OF OXIDE SCALES TO ENERGETIC PARTICLE IMPACT: INITIAL RESULTS AND EVALUATION OF EXPERIMENTAL TECHNIQUE*

P. F. Tortorelli and J. R. Keiser

ABSTRACT

The mechanical response of preformed oxide scales to energetic particle bombardment is being studied in a modified scanning electron microscope. Initial results showed that cracking can occur from single-particle impacts and that local deformation of the scale is sensitive to the presence of and the method used for producing the oxide and to the composition of the target alloy. Preliminary experiments with Fe–20Cr–25Ni–0.7Nb and Fe–20Cr–25Ni–0.7Nb + 0.13Ce steels revealed no effect of the reactive element addition on the cracking propensity of the oxide scale. However, the accommodation of the scale and underlying metal to the strain caused by impact varied for different concentrations of yttrium in Ni–20Cr–12Al. Thin oxide scales on Ni–20Cr–12Al–0.1Y were observed to deform compliantly around the edges of the craters formed on impact. Data for the Ni–20Cr–12Al–Y alloys suggested that the yttrium can impart increased resistance to scale deformation under certain conditions. The current technique for evaluating the impact response of oxide scales therefore appears to offer promise as a way to characterize oxide scales and to gain information about the validity of certain mechanisms that deal with the role of reactive element or dispersoid additions in improving scale adherence. The potential of the technique can be better evaluated when additional characterization procedures, including in situ observations of crack growth and healing, are included.

1. INTRODUCTION

Protection of metals and metallic alloys from degradation by high-temperature corrosion normally requires a compact, compliant, adherent surface layer that is either inert to, or slowly reactive with, the environment. This surface layer can be a coating or a corrosion product (normally an oxide) that forms upon initial exposure. (In most practical high-temperature applications, this layer is either alumina or chromia.) Because of their importance in protecting the underlying metal from

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attack, much research and development have been conducted on the growth, stability, and adherence of such oxide scales.

Under aggressive corrosion conditions and/or due to the influence of thermal cycling or erosion, many oxide scales fail to provide protection because of cracking and spallation. However, it is now well established that relatively small concentrations of rare-earth (and other) oxide dispersoids or reactive elements in certain alloys can promote the formation of more adherent oxide scales (see the reviews of dispersoid/reactive element effects in refs. 1–4). Examples of such additions include yttrium in alumina- and chromia-formers[5-13] and ThO_2, Y_2O_3, CeO_2, and Al_2O_3 in Ni–20 wt % Cr.[14,15] While there is no doubt that these reactive element/dispersoid (RE) additions generally improve scale adherence, there is considerable debate regarding the mechanism(s) by which such an effect occurs. Proposed mechanisms[1-5,12,13] include oxide pegging, vacancy annihilation, modified growth processes that decrease stresses in the scale, enhanced scale plasticity, and improved chemical bonding in the presence of REs through their influence on bond strengthening and/or on gettering of indigenous impurities that otherwise weaken the adherence of the oxide scale to the underlying metal.

This report describes the first observations of what happens to preformed oxide scales when they are subjected to microscopic impacts. The purposes of such experiments are to better understand how scales accommodate strain in terms of cracking and deformation and to determine what effect RE additions may have on this response. The information can then be used to judge the applicability of proposed mechanisms of scale adherence that deal with enhanced scale plasticity or toughening due to the presence of reactive elements or rare-earth dispersoids. Preliminary data for thin scales on prototypic chromia- and alumina-formers are presented, and an initial evaluation of the Impact Response of Oxide Scales (IROXS) technique is made.

2. EXPERIMENTAL PROCEDURES

The IROXS technique involves the use of a unique experimental system that was designed principally for studies of erosion and corrosion-erosion.[16] The instrument consists of a modified scanning electron microscope (SEM) that incorporates a particle gun, hot stage, and preheated gas delivery system into its specimen chamber. The gun fires tungsten carbide spheres (0.34-mm diam) at a target that can be heated to 1100°C. The SEM and other components of the experimental system have been described in detail elsewhere.[16]

The study of the response of oxide scales to energetic particle bombardment employs the above-described system to impact preformed oxide scales on a microscopic level and then to characterize the impact response in terms of (1) the propensity for deformation and cracking, (2) crack growth, and (3) the tendency for cracks to heal. At this stage, the results consist of only observations of deformation and cracking propensity. Furthermore, while impacts can be made at different incident angles and at any temperature up to 1100°C, experiments have thus far been conducted at normal particle incidence and ambient (22–25°C) temperature.

The initial experiments were performed with alloys of two different compositions that allowed examination of both chromia- and alumina-forming materials. The chromia-forming alloy is an Fe–20Cr–25Ni–0.7Nb (wt %) steel (denoted below as 20/25/Nb steel) with and without 0.13 wt% Ce additions and was obtained from Harwell Laboratory, United Kingdom. The other alloy is wrought Ni–20Cr–12Al (wt %) containing 0.05–3Y and was provided by United Technologies Research Center, East Hartford, Connecticut. The two-phase Ni–20Cr–12Al consists of βNiAl and γNi solid solution.[17] Both alloy types were preoxidized in air for 30 min at 930°C before they were inserted into the microscope chamber for subsequent impact. This oxidizing treatment does not result in fully mature scales, particularly in the case of the Ni–20Cr–12Al alloys, where only very thin oxide scales were selectively formed (see Fig. 1). Nevertheless, as shown below, the presence of such scales affected impact response under certain conditions.

3. RESULTS

3.1 Fe–20Cr–25Ni–0.7Nb STEEL

Two specimens each of 20/25/Nb steel and 20/25/Nb + 0.13Ce were examined in the SEM. In all four cases, regular circular impact craters were observed. Examples of such craters are shown in Fig. 2. The results are summarized in Table 1, in terms of crater diameter and visual evidence of cracks. Most cracking was circumferential, although some radial cracks were also present (see Fig. 3). The results on cracking do not strongly

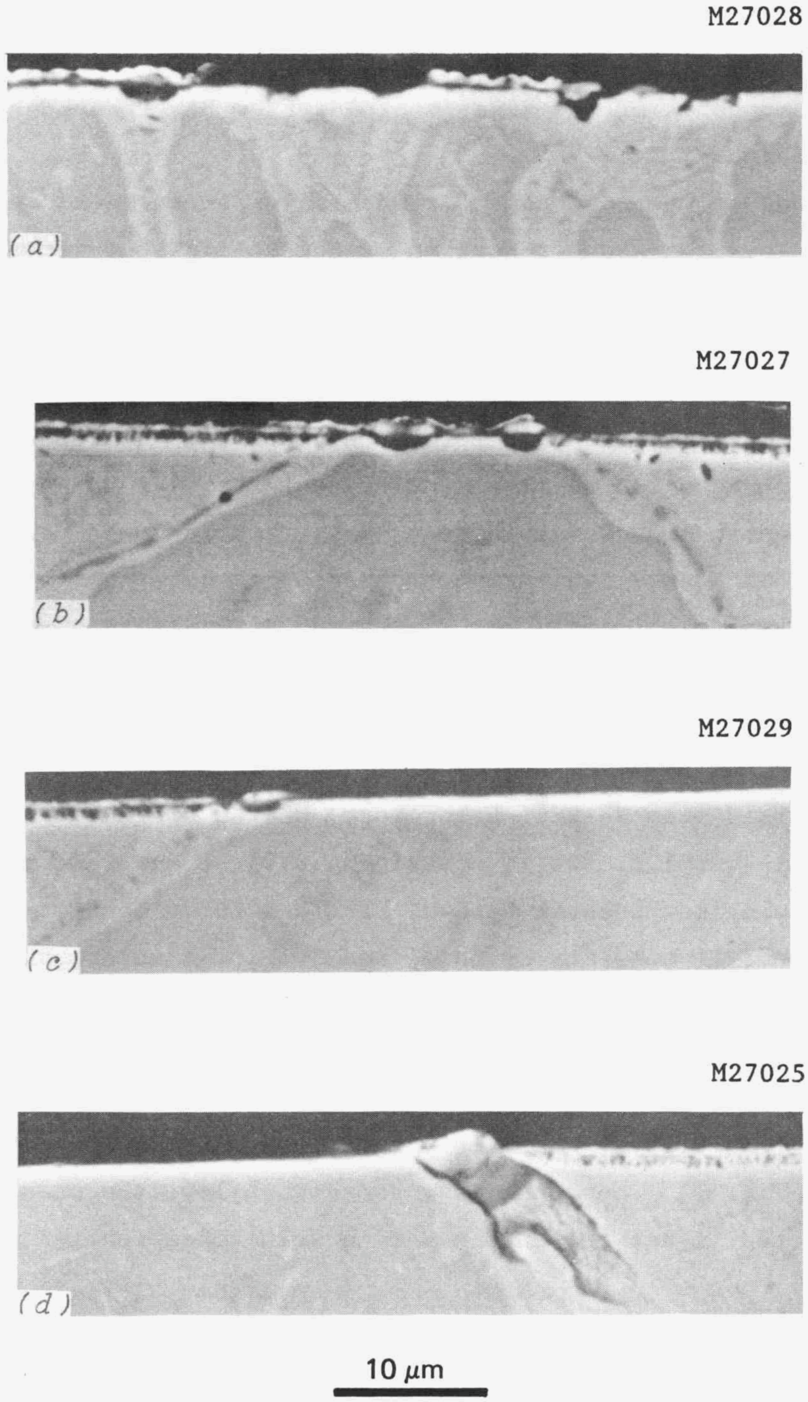

Fig. 1. Polished cross sections of Ni–20Cr–12Al alloys oxidized in air at 930°C for 30 min. ***(a)*** 0.05Y. ***(b)*** 0.1Y. ***(c)*** 0.5Y. ***(d)*** 3Y.

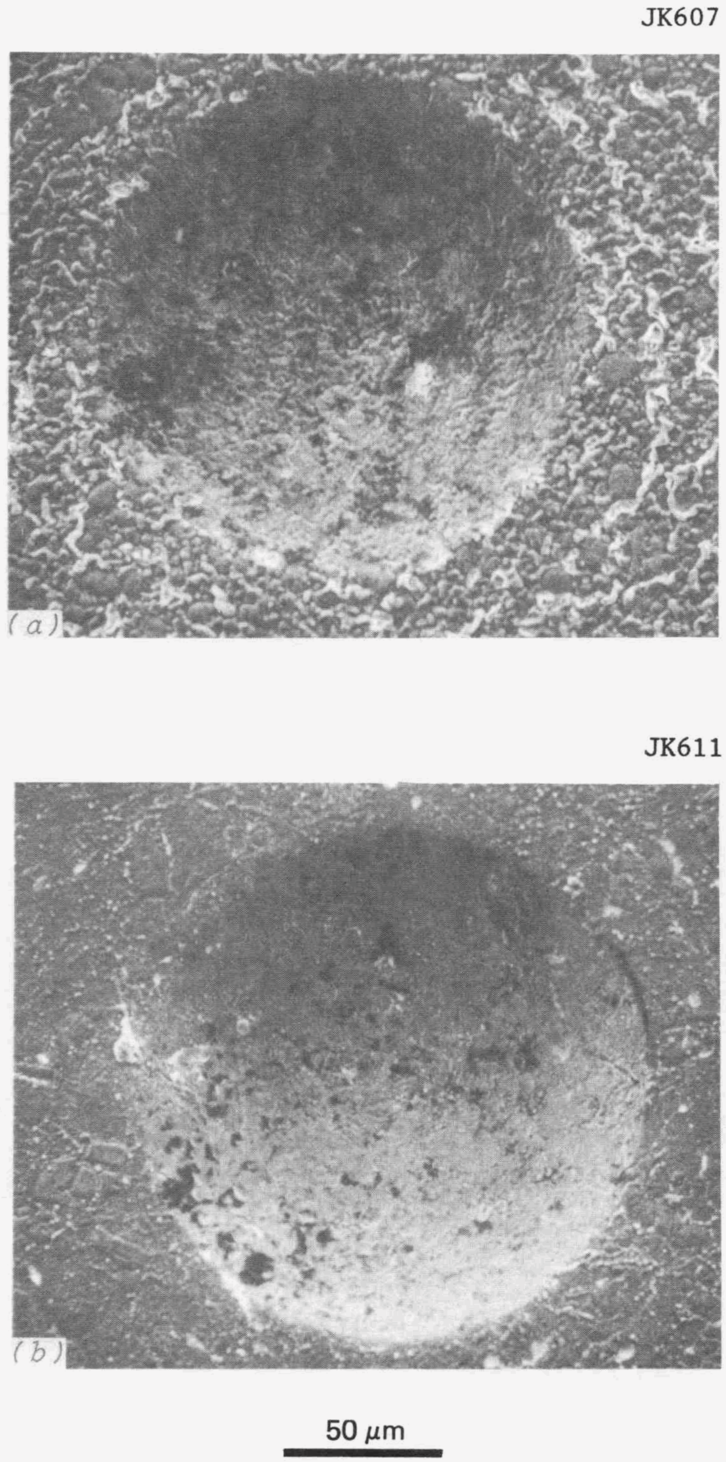

Fig. 2. *(a)* Oxidized Fe–20Cr–25Ni–0.7Nb steel impacted at 44 m/s. *(b)* Oxidized Fe–20Cr–25Ni–0.7Nb + 0.13Ce steel impacted at 37 m/s.

Table 1. Diameters of craters formed on oxidized 20/25/Nb steel by particle impact and observations of cracking

Material	Particle velocity (m/s)	Crater diameter (mm)	Observed cracks
20/25/Nb	27	0.17	Yes
20/25/Nb	44	0.15	No
20/25/Nb	61	0.23	Yes
20/25/Nb + 0.13Ce	25	0.10	No
20/25/Nb + 0.13Ce	37	0.16	No

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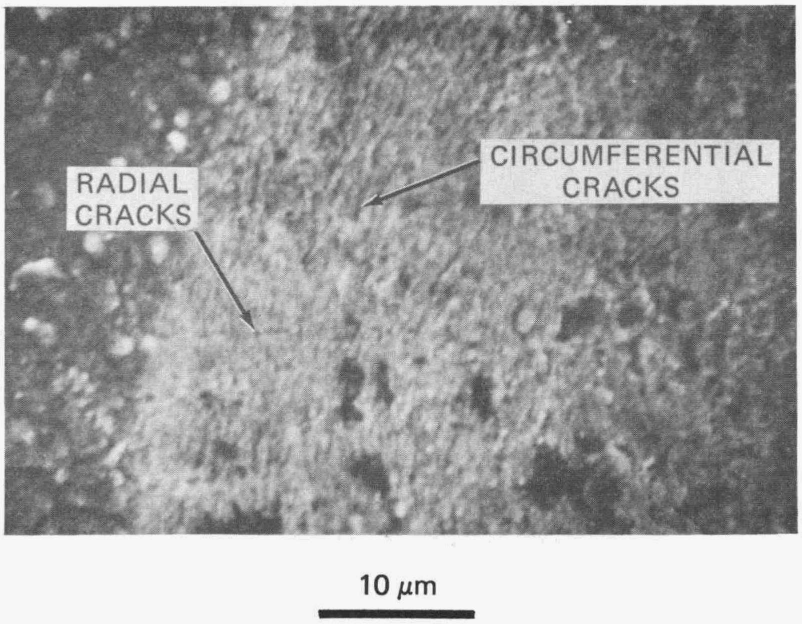

Fig. 3. Oxidized Fe—20Cr—25Ni—0.7Nb steel impacted at 27 mm/s.

indicate that the presence of cerium prevented cracking of the oxide scale, particularly in view of other observations for higher (but unknown) impact velocities that revealed cracking of the scale formed on a 20/25/Nb + 0.13Ce steel.

3.2 NiCrAlY

The Ni-20Cr-12Al alloys form a compact alumina scale when they are oxidized above 1000°C. However, for this scale to provide protection, it must retain its adherence to the underlying metal as it thickens and under thermal cycling. However, such adherence is lacking in this alloy system unless yttrium (or another selected element or dispersoid) is present. Initial impact experiments with this alloy system used specimens that were oxidized for short times at 930°C. Observations of the response of these thin oxide scales to energetic particle impact were then made as a function of yttrium content. Representative SEM photographs of craters caused by such impacts are shown in Fig. 4. (Note that the surfaces were only partially oxidized as a result of the low-temperature, short-time oxidizing treatment.) The most striking observation was that most of the craters had irregular peripheral outlines despite impact by a spherical particle. Indeed, the only circular craters were those formed on the two specimens with 0.1Y; those on oxidized Ni-20Cr-12Al containing either less (0.05%) or more (0.5, 1, or 3%) yttrium had irregular boundaries.

More detailed examination of the two specimens containing 0.1Y revealed differences between them. In one case, the impact seemed to break through the oxide and damage the underlying metal (Fig. 5). On the other 0.1Y specimen, the craters were shallower and the oxide film deformed over the edges of the craters without cracking or detachment from the underlying metal (Fig. 6). On the latter specimen, the type of deformation was similar for both craters despite quite different measured impact velocities [29 m/s for that shown in Fig. 6*(a)* and 76 m/s for that in Fig. 6*(b)*]. The crater diameter was larger for the specimen formed at higher particle velocity.

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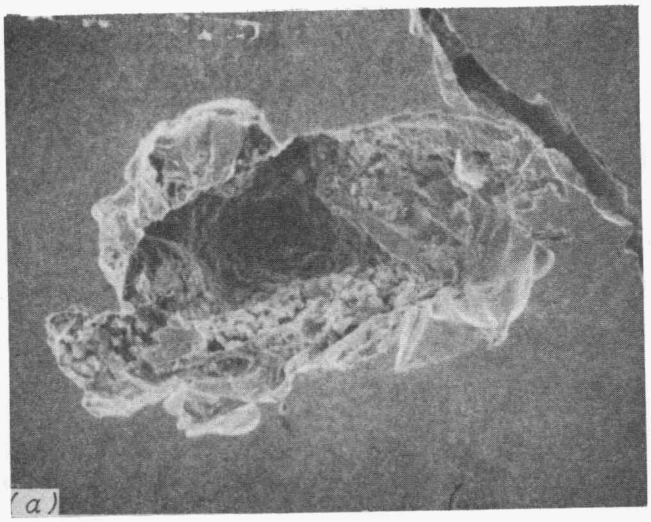

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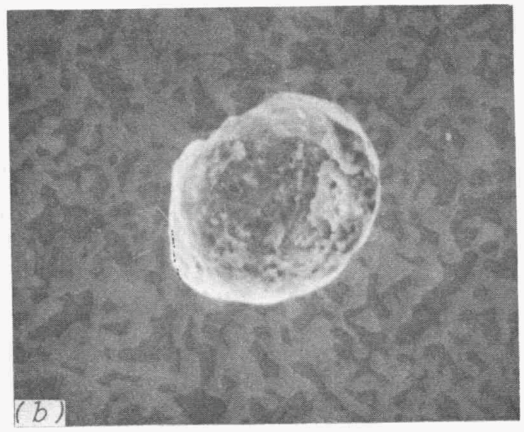

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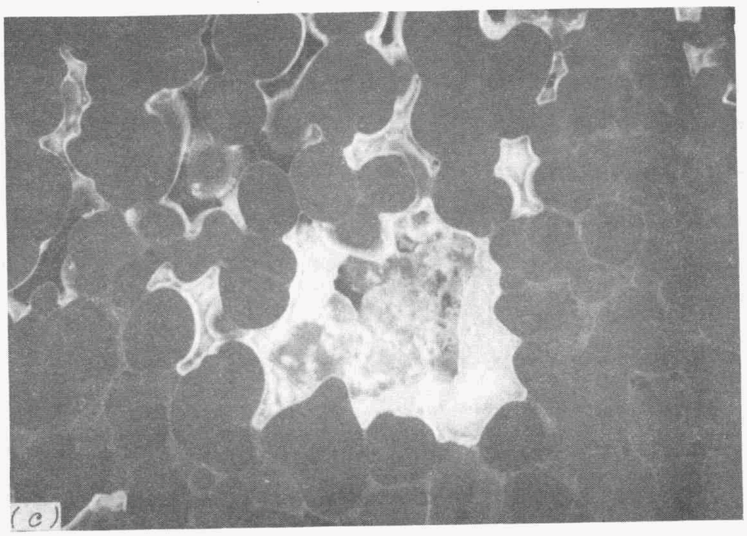

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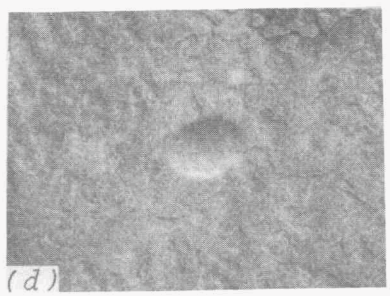

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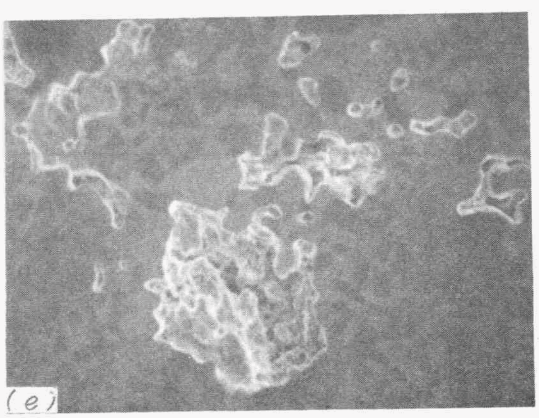

300 μm

Fig. 4. Oxidized Ni–20Cr–12AlY alloys. *(a)* 0.05 wt % Y, impacted at 35 m/s. *(b)* 0.1 wt % Y, impacted at 30 m/s. *(c)* 3 wt % Y, impacted at 29 m/s. *(d)* 0.1 wt % Y, impacted at 33 m/s. *(e)* 0.5 wt % Y, impacted at 32 m/s.

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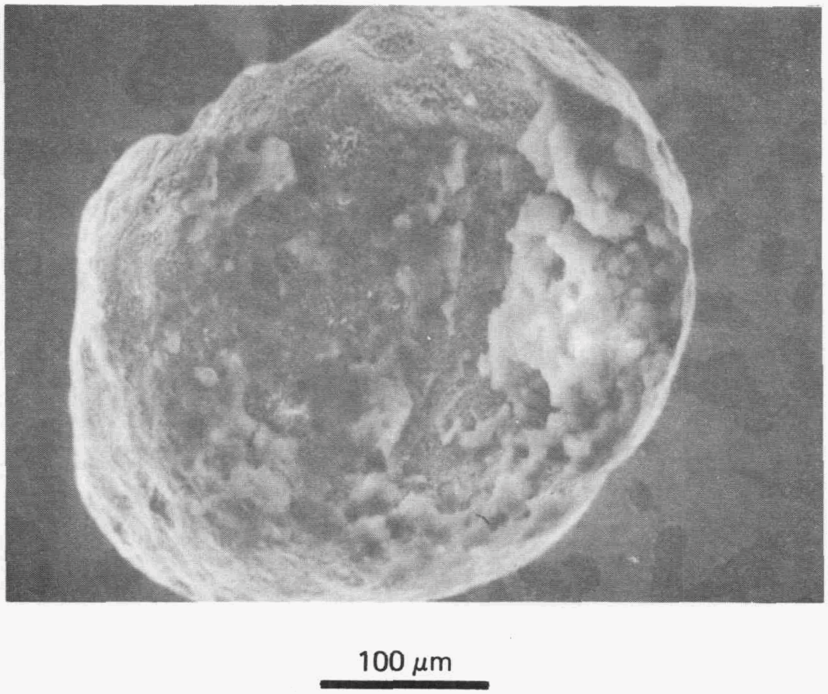

Fig. 5. Oxidized Ni–20Cr–12Al–0.1Y impacted at 30 m/s.

4. DISCUSSION

4.1 SIGNIFICANCE OF RESULTS

Because of the early stage of this investigation and the limited number of specimens examined, only prelimimary conclusions can be drawn. However, the above results reveal some rather interesting and potentially important information about the subject oxide scales. The impact response of the chromia scale formed on the 20/25/Nb steels differs considerably from that of most of the mixed oxide scales formed on the NiCrAlY alloys. This finding is significant because it shows that the impact response is sensitive to the type of oxide scale present on the alloy. Another important result of these preliminary experiments is the demonstration that the amount of yttrium in the NiCrAlY alloys has an effect on the response of these materials to impact. Specifically, it appears that the presence of an optimal yttrium concentration imparts a plasticity to the scale that does not occur in the absence of this element[5] or when it is more highly concentrated to the point where resistance to deformation is possibly increased.[18]

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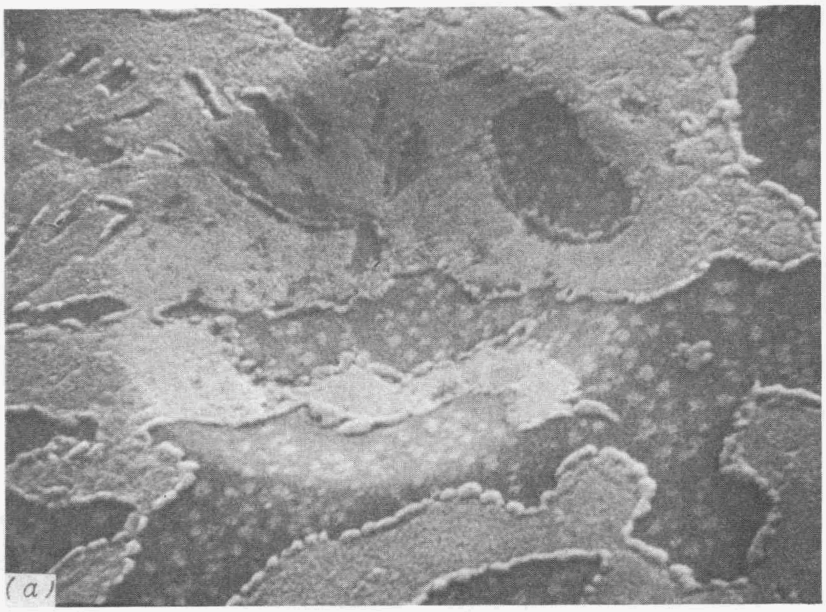

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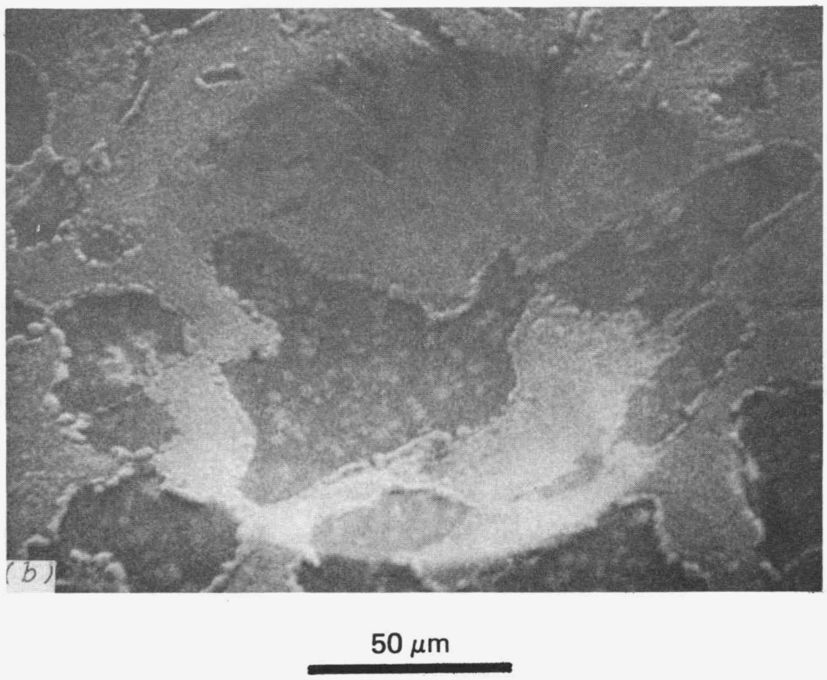

Fig. 6. Oxidized Ni–20Cr–12Al–0.1Y. *(a)* Impacted at 29 m/s. *(b)* Impacted at 76 m/s.

The irregular crater outlines observed for the higher yttrium concentrations may be indicative of the higher deformation resistance of the selectively formed oxide grains, which yield less than the remainder of the surface material. However, such a conclusion about the influence of an RE as a function of its concentration must be confirmed by additional data on both unoxidized and oxidized materials. It is interesting to note that the irregular crater boundaries have never been observed previously for similar impacts on unoxidized metals and alloys[16,19,20] and that, in the current experiments, impacts on unoxidized Ni–20Cr–12Al containing 0.5Y and 3Y resulted in circular craters (see Fig. 7). The presence of the thin scales on these alloys significantly affected the impact response.

The observed deformation of the oxide scale on the two NiCrAl-0.1Y specimens (Fig. 6) is also significant: the ability of such a scale to deform in conformance with the underlying metal attests to a fundamental plasticity not often associated with oxides. However, this ductility may be partly associated with the thinness of the oxide, and the practical importance of this phenomenon awaits data on the response of thicker and more compact alumina scales that form upon oxidation for longer periods or at temperatures above 1000°C.

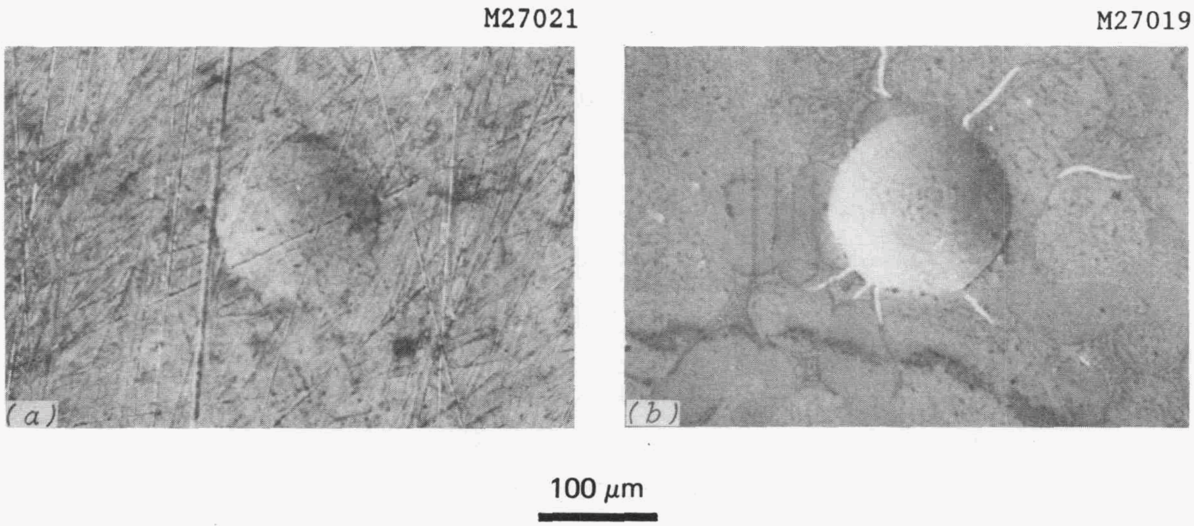

Fig. 7. Impact craters formed on unoxidized Ni–20Cr–12Al. ***(a)*** **0.5Y.** ***(b)*** **3Y.**

The data for the 20/25/Nb specimens do not indicate that the presence of a reactive element addition definitely affects the ability of a preformed oxide scale to resist cracking. Therefore, it does not appear that, at least in this case, the reactive element addition "toughens" the oxide scale in the sense of substantially reducing the nucleation of or propenity for cracking. However, there may be other effects induced by this RE, specifically those associated with resistance to further crack growth or enhanced healing of cracks, that may be revealed in the next phase of this project (see below).

An important aspect of the comparison of the impact response of scales formed on alloys with and without "reactive" element additions is the necessity of evaluating "equivalent" scales.* Because of the dramatic effects of RE additions on scale growth kinetics and microstructure, the scale resulting from a given oxidation treatment for alloys with RE will be different from those without RE. Examples of this difference were found in the current study, where the amount of retained alumina tended to vary among the different compositions of the NiCrAlY. Similarly, differences in scale morphology (as seen from the micrographs in Fig. 2) and thickness can be found between 20/25/Nb and 20/25/Nb + 0.13Ce steels oxidized under the same conditions.[11] Therefore, it is important to discuss the response of oxide scales to energetic impact in terms of both direct and indirect effects of REs and to use, when possible, specimens with preformed scales of equivalent thickness by variation of the oxidizing treatment.

4.2 EVALUATION OF TECHNIQUE

The experimental data served to demonstrate a principal required condition for the IROXS technique: that "microscopic" cracking of oxide scales can be induced by the use of high-velocity spherical particles. More importantly, our preliminary findings showed that even relatively unsophisticated examination of the impact response of preformed oxide scales reveals some sensitivity to the presence of scales and to compositional differences. The IROXS technique thus has potential for furthering our

*Personal communication from M. J. Bennett, Harwell Laboratory, Oxfordshire, United Kingdom, to P. F. Tortorelli, Oak Ridge National Laboratory, Oak Ridge, Tennessee, July 1987.

understanding of the influence of certain RE additions on scale characteristics. The preliminary data will be of more value when combined with those from the additional (and more difficult) characterizations to be included in the impact response experiments that will be conducted in the next phase of this study. These will include in situ observations of crack growth (and perhaps spallation) during rapid cooling in the SEM and of crack healing while an impacted specimen is held at temperature. When systematically made as a function of RE additions to chromia- and alumina-formers, these observations can contribute to the development of appropriate mechanisms of scale adherence in systems of importance to high-temperature applications. As such, even negative results, that is, no effects of additions on cracking or deformation of impacted scales, can be of value in judging the merits of particular models.

The attractiveness of the IROXS technique is that it is, in principle, relatively simple and straightforward (apart from the equivalent scale complication discussed above) and yields results that relate directly to the accommodation of strain by oxide scales. However, the technique's advantages and uniqueness of approach have to be considered in light of the atypical source of stress (particle impact) and the various experimental problems, which include the quantification of cracking propensity, the difficulty of locating and imaging craters (particularly irregular ones), and the apparent lack of correlation of crater size with measurements of particle velocity. This latter problem, however, may be related to the measurement difficulties associated with small projectiles moving at high speeds[16] and may not be indicative of an inconsistent material response to impact. Despite these drawbacks, the use of microscopic impact response seems to hold promise as a new and different way of acquiring information on the characteristics of oxide scales and of gaining insight into the mechanisms by which they remain adherent to the underlying metal.

5. FUTURE WORK

The next series of experiments using the IROXS technique will involve impacts made as a function of temperature on specimens that have well-characterized and more fully developed oxide scales. This plan not only follows from natural experimental progression, but is also intended to address the important "equivalent scale" considerations discussed above. Furthermore, the full potential of the technique will be explored by evaluation of two other methods of characterization of the cracks that may form upon impact. One method is the observation of crack growth in the SEM during cooling of the specimen from impact temperature (if a sufficiently large ΔT can be induced) to investigate whether RE additions affect this stage of cracking. The second method involves conducting experiments on cracked scales in which the specimen is held at elevated temperature in the SEM after impact; any tendency for crack healing is monitored in situ in the presence or absence of an oxidizing species. Such observations can directly test a model in which RE additions exert a positive influence on scale adherence by promoting the healing of cracks created by growth stresses. Indeed, it is known that crack healing can occur below a critical strain rate[21] and it is interesting to speculate on whether REs can enhance this process and thus reduce corrosion and improve scale adherence.

6. SUMMARY

Initial results from the study of the response of preformed oxide scales to energetic single-particle bombardment showed that cracking can be induced by such impacts in a modified SEM and that the local deformation was sensitive to the presence of the scale and the composition of the oxidized target alloy. Preliminary experiments with Fe–20Cr–25Ni–0.7Nb and Fe–20Cr–25Ni–0.7Nb + 0.13Ce steels revealed no tendency for the reactive element addition to modify the cracking propensity of the oxide scale. The accommodation of the oxide scale to the strain caused by impact varied for different concentrations of yttrium in Ni–20Cr–12Al. Thin oxide scales on Ni–20Cr–12Al–0.1Y were observed to compliantly deform around the edges of the craters formed on impact. Data for the Ni–20Cr–12Al–Y alloys suggest that the yttrium can impart increased resistance to scale deformation under certain conditions.

The IROXS technique appears to offer promise as a way to characterize oxide scales and to gain insight into the validity of certain mechanisms that deal with the role of reactive element or dispersoid additions in improving scale adherence. Its full potential can be evaluated when additional characterization procedures, including in situ observations of crack growth and healing, are examined. When used with data on crack healing tendencies, the IROXS technique offers a direct test of a model in which reactive elements or dispersoid additions to the metallic alloy promote healing of cracks in the scales and lead to better scale adherence and reduced corrosion.

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