Durability Testing of Ceramic Coatings for Indirect Resistance Heat Treating in Vehicle Lightweighting Applications

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Abstract

The need for vehicle lightweighting has driven the development of new component designs, materials, and thermal processing of materials. One approach has been the development of technology that allows efficient, localized material property modification through the direct or indirect heat treatment (DRHT and IRHT) processes. These approaches provide an effective means of producing localized material property modifications without the need to heat the overall sheet. This decreases the cost of materials, improves cold formability, and maintains strength in the overall component. Conventional resistance welding equipment has been demonstrated and utilized in these approaches. This paper describes the fundamentals of the IRHT processing, which uses a set of resistively heated molybdenum-based titanium zirconium molybdenum (Mo-TZM) elements that are electrically isolated from the workpiece. The TZM elements are ceramic coated using thermal spray methods to enable the thermal cycles required to heat treat specific areas of the workpieces. This paper details a program to evaluate the durability of different high-temperature ceramic coatings exposed to temperatures in excess of 1100°C. Candidate ceramics were based on zirconia and alumina, and their combinations. These ceramics were selected based on electrical conductivity, coefficient of thermal expansion (CTE) compatibility with the Mo-TZM electrodes, minimal interaction with oxygen, and applicability for economical thermal spray technology application.

The IRHT heating system will be described, along with a dynamic, resistance-based method to measure the temperature of TZM elements. The test results showed different modes of ceramic coating durability and failure modes. The baseline, yttria-stabilized zirconia (YSZ) coating, failed by progressive coating removal across element face, the Al_2O_3/ZrO_2 coating failed by progressive spallation of the coating, and the mullite coating failed as a result of overheating the TZM/mullite interface. A discussion of the coating degradation characteristics includes the thermal, chemical, and mechanical (fixture) related causes of degradation. It was concluded that the Al_2O_3/ZrO_2 coating provided the best overall endurance, followed by YSZ, and mullite without a bond coat. All of these ceramic systems may provide acceptable performance if the thermal management of the element is improved (addressing the fixture and coating spallation issues). Further improvements to the IRHT system can be obtained by improving the ceramic coating adhesion and toughness, redesigning the heating element shape, and improving the thermal management of the fixture. The present work and implementation of some of these improvements should allow the IRHT process to produce localized material properties that will reduce material and processing costs in the lightweighting of automotive structures.

Introduction

The need for automotive vehicle lightweighting for improved fuel economy has led to a number of new materials and innovations. A new generation of advanced high-strength steels (AHSS) steels has emerged (e.g., “Gen 3” steel) with improved formability and high strength. There is also renewed examination of magnesium and other lightweight metals. Typically, commercially available materials are used in their as-received condition. It is common to weld additional components to a structure to impart local stiffness or other property enhancements enabling improved serviceability for a particular application. However, in some cases, local metallurgical alterations to the material itself can produce the desired engineering properties without the need for incurring the added costs associated with attachments. Possible base metal alterations include localized and configured hardening, softening, grain refining, or metallurgical transformations that can provide useful hybrid properties to the component. One method of producing localized metallurgical alterations is through the use of indirect resistance heat treating.

IRHT is a method of locally tailoring metallurgical or physical changes in a workpiece by the indirect conduction of heat from a set of electrically isolated, but thermally conductive resistive heating elements followed by rapid quenching. The method has previously been demonstrated for indirect resistance brazing.[(1)] A sample heated using this technology is shown in Figure 1. High electrical currents are used to generate resistive heat within the metallic heating elements of a specified geometry. A ceramic coating on the heating element acts as an electrical barrier, but allows heat transfer to and from the workpiece. Water-cooled copper tooling is used to position the heating element, provide electrical contact, and cool the element to prevent overheating. On cessation of current flow, heat in the workpiece flows back through the ceramic coating into the heating element, and to the copper tooling. A schematic of this process is illustrated in Figure 2. The resulting thermal cycle provides the desired metallurgical or physical transformations. The process is closely related to DRHT, described elsewhere.[(2)] Laboratory experiments have illustrated the capability to produce locally hardened zones with appropriate microstructures. Locally hardened zones are used to strengthen components where needed, enabling a reduction in overall thickness. Locally softened zones are also possible, creating crumple zones in crash energy management systems.

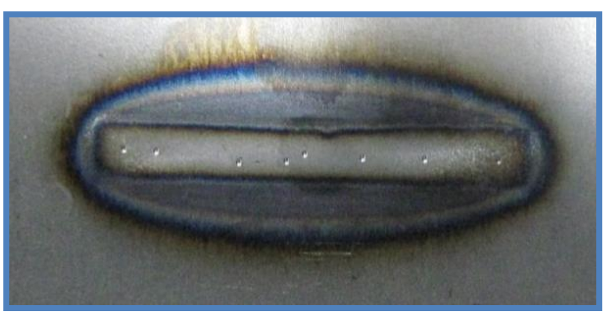

Figure 1. DP780 Sheet Steel Sample Following Local Indirect Resistance Heat Treatment

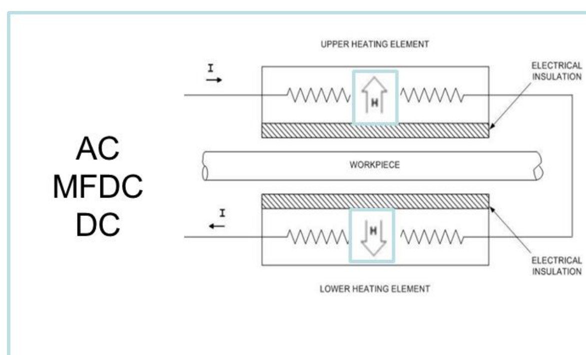

Figure 2. Schematic Illustration of the Indirect Resistance Heat Treating Process

An important consideration in the design of the IRHT system is the material selections for both the heating element and the ceramic coating. The heating element must be refractory in nature and able to withstand high temperature exposure to oxygen. The ceramic coating(s) should have high electrical resistance, high thermal stability, good toughness, good adherence to the substrate, have metallurgical and CTE compatibility with the heating element, and have stability when exposed to oxygen at high temperatures. The coated heating element must have extended durability to be viable at the high temperatures required for most heat treatment applications. However, the availability of ceramic coatings applied to the heating elements for high temperature applications (heating temperatures well in excess of 1000°C) are limited. Further, the present ceramic coatings used for high temperature IRHT trial applications have shown thermal degradation and consequently, limited life. This study evaluates the durability of alternate ceramic coatings for high temperature applications, such as IRHT.

Heating Elements for Study

In this study, Mo-TZM electrodes have been used as the resistive heating element material for these trials. The IRHT elements shown in Figure 3 were developed for heating an extended area (10×50 mm) of sheet metal samples.

Figure 3. An Example of an Ceramic Coated TZM Heating Element for the Indirect Resistance Heat Treatment Process

For this application, the ceramic coating required:

(1) Durability to exposure to an oxygen-containing environment at temperatures up to 1400°C.
(2) CTEs compatible with Mo-TZM.
(3) Good thermal conduction allowing heat flow from the element to the substrate.
(4) Dielectric properties, effectively electrically insulating the element from the substrate.
(5) Toughness at both high and low temperatures for durability.
(6) Easy application to the Mo-TZM element.

Sprayable plasma coatings for high (>1000°C) or very high temperature (>1800°C) use are, however, limited. Many of these coatings have incompatible CTE matches to TZM, are susceptible to oxidation, are very expensive, are not readily sprayable, or have other drawbacks that discourage their use in the present application.

The coatings for accelerated durability testing in this program were selected as:

- Mullite thermal barrier coating (TBC) without a bond coat.
- Mullite TBC with a bond coat.
- Al_2O_3/ZrO_2 TBC with a bond coat.
- YSZ/AlN TBC with a bond coat (baseline).

The bond coating in this case was a layer of 0.25-mm NiCr. The NiCr coating improved coating adhesion, accommodated thermal CTE mismatches between the TZM and the YSZ coating, and discouraged Mo sublimation from the TZM. The baseline ceramic coating thickness for all of the top coat coatings, including the baseline coating was a 0.5-1.0 mm. The spray parameters for the YSZ/Al were similar to those used for YSZ. The applied plasma spray (APS) parameters of the other coatings were developed appropriately. After coating, the surfaces were ground flat.

The work material in the present application is 0.9-mm DP780 uncoated steel.

Details of the IRHT System

The basic design of the IRHT tooling is provided in Figure 4. This tooling provided a series current path between two opposing IRHT heating elements. The tooling also included direct water cooling of copper support tooling under each heating element. This tooling was then placed within a standard 100-kVA AC pedestal resistance welding system. An image of one heating element as constrained by the tooling is provided in Figure 5.

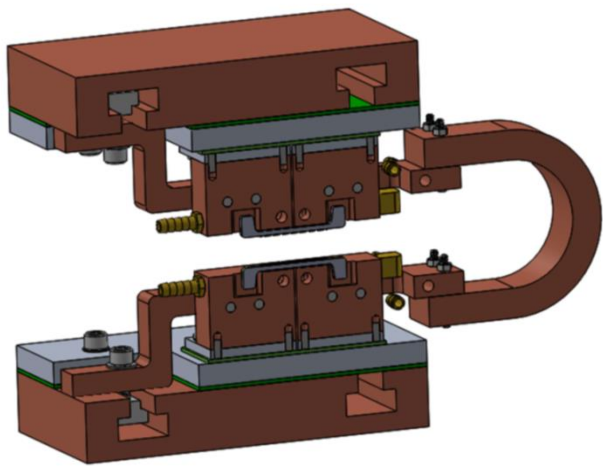

Figure 4. Schematic Cutaway Section of the IRHT Tooling Used in This Investigation

Figure 5. The Coated Mo-TZM Heating Element Positioned by the Tooling in the Pedestal Welding Machine

Measurement of the IRHT process temperature was done by collecting the dynamic resistance of the TZM elements themselves. For the TZM material, temperature varies linearly with resistance. Temperature-resistivity data for Mo-TZM is provided in Figure 6. The temperature was correlated against both attached thermocouples and thermal imaging. Dynamic resistance was calculated by monitoring the current and voltage across a heating element (voltage lead placement shown in Figure 5) and calculating the resistance at the instantaneous peak of the current waveform. The temperature profile of the TZM elements during heating was adjusted using a pulsation schedule. This facilitated profiles that heated rapidly and remained nominally steady over the desired processing time. An example of a 33-s heating cycle is shown in Figure 7. This graph shows the temperature readings from both the thermocouple and the IR camera overlaid with the AC current (represented by a voltage reading). Resistances during cooling were assessed by periodic monitoring of small, dispersed pulses of low current. This again was compared to the values observed using a thermocouple.

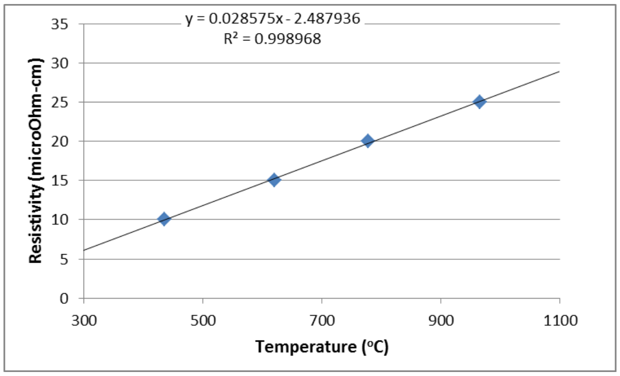

Figure 6. Linear Relationship between Electrical Resistivity and Temperature for TZM Material

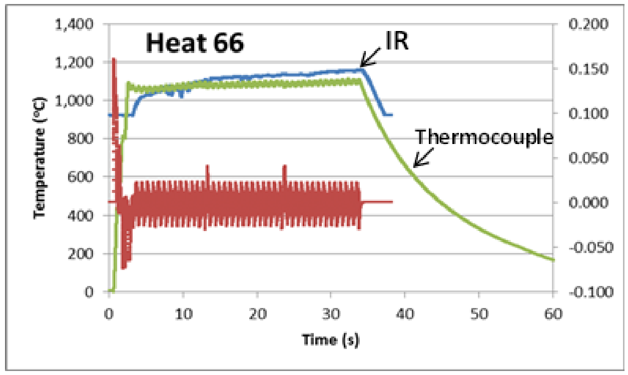

Figure 7. Temperature Measurements from Both IR Camera and Thermocouple Overlaid with the AC Current Pulsation Profile for a 33-s Heat Treatment

In-situ Visual Inspection of Heating Element Contact Faces

Monitoring of heating element face conditions was done using a set of mirrors combined with a digital camera. The mirror system allowed photographing of upper and lower faces of the ceramic at specific intervals during the durability testing. This unit is shown in Figure 8. The camera was placed on a tripod and located in the identical position to repeatable imaging. Figure 9 shows an example of top and bottom electrode faces and two of the sides at the termination of a test. Independent pictures of the element faces (loose from the fixture) are also shown for comparison.

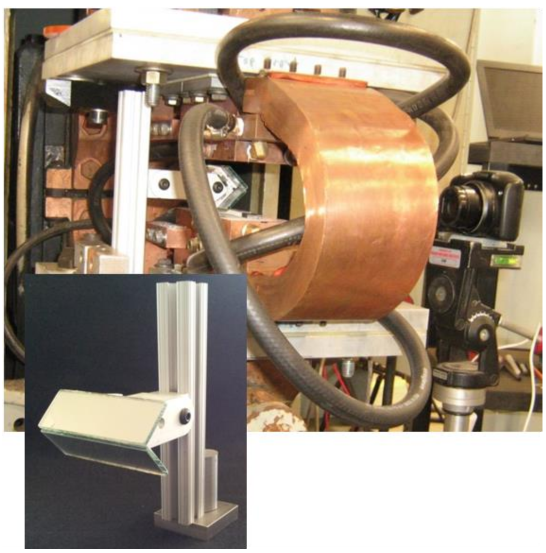

Figure 8. Apparatus for In-Situ Photographing Opposing Electrode Faces (insert shows the double mirror used to simultaneously photograph top and bottom heating elements)

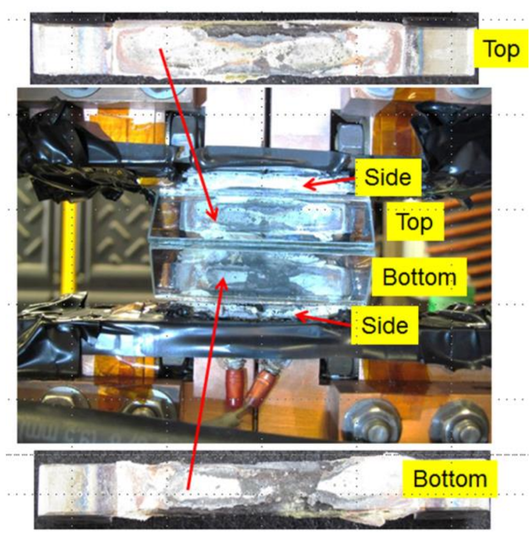

Figure 9. Example of Electrode Face Images Taken during Endurance Testing (superimposed are top and bottom photographs taken from the same set of electrodes after removal from fixture)

Experimental Endurance Trials

Endurance testing was done by repeatedly applying areas of heat treatment on extended strips of steel. Localized heat treatment can be as short as a second or two in practice.[2] However, to abbreviate the durability test, individual thermal cycle pulses including 45 s of heating and 45 s of cooling were employed. The current pulsation sequence was configured to achieve and hold the target temperature over that time. Tests at various electrode forces were used to evaluate the durability of the ceramic coating.

Measurements made in the durability testing included: dynamic current and voltage for each heating trial, visual inspection of ceramic coating degradation (photographs of element face) every 10 heating cycles, as well as qualitative visual assessments of degree of contact between the element and the steel. Tests were conducted up to 100 heating cycles or when excessive wear on electrodes was observed. An example of a durability panel is shown in Figure 10. Excessive wear was defined as a complete breach of coating from edge to edge across the face, gross spallation of the coating, gross exposure of the base coating in contact with the TZM element, or when melting of the steel sheet occurred.

Figure 10. Example of an IRHT Durability Test Plate in This Program

Two tests of elements using each coating type were performed at the nominal aim temperature of 1100°C. These two tests were performed at differing forces with the intention of improving thermal contact between the electrode and the steel. A table of test parameters and results is given in Table 1. Figure 11 shows the surface conditions of areas heat treated under the differing conditions of these tests. The data provided in this figure includes the trial designation and the numbers of hits to failure (number of thermal cycles) for the different coatings and force levels.

Table 1. Summary of Test Results Showing Test Pulsation Conditions, Applied Force, and Aim Temperature

ID	Coating	Applied Force (kN)	Aim Temp (°C)	Hits to Failure	Element Contact
G	YSZ	10.0	1100	100	OK
D	YSZ	3.8	1100	12	Very good
B	Al_2O_3/ZrO_2	3.8	1200	96	Very good
A	Al_2O_3/ZrO_2	3.8	1100	100	OK
E	Mullite NB	10.0	1100	70	OK
V	Mullite NB	3.8	1100	40	Good
C	Mullite BC	3.8	1100	32	Very good
H	Mullite BC	10.0	1100	14	Very good

Note: NB – No bond coat
BC – Bond coat

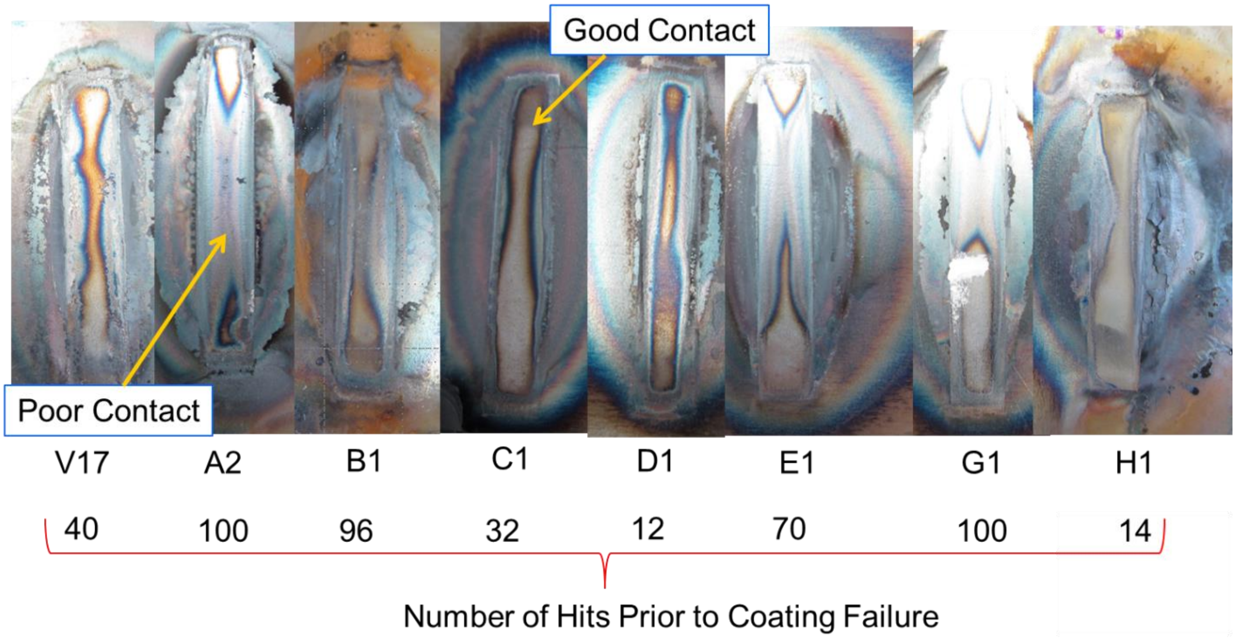

Figure 11. Surface Quality and the Numbers of Cycles to Failure for the Various Trials Conducted in This Study (images shown highlight differences in contact between the electrodes and the steel)

Durability Test Results

The durability testing of the baseline YSZ ceramic coating (Series G) was conducted to the limit of 100 cycles. Progressive coating removal across element face was noted. The low force variant of the YSZ coating (Series D) was terminated after only 12 thermal cycles due to excessive heating and melting at the center of the steel strip. This was accompanied by a decrease in the measured temperature over these thermal cycles. The Al_2O_3 ceramic coating (Series A) tested at low force failed by excessive wear produced by progressive spallation of coating from the face after 100 thermal cycles. A high-temperature variant failed after 98 hits due to cracking of the TZM. In both cases, the temperature of the element increased toward the end of the durability testing. The mullite coating without a bond layer (Series E) tested at high force failed by overheating at the TZM/ceramic interface after 70 thermal cycles. This resulted in the melting of steel under the heating element. The low-force variant (Series V) failed after 40 cycles due to progressive spallation across electrode width. Mullite with a bond coat at low force (Series C) failed at the TZM/bond coating interface after 32 heating cycles. The high force variant (Series H) failed after 14 thermal cycles by gross spallation of the mullite from the base coat.

Discussion

The results presented above suggest that overall, the best durability was experienced by the Al_2O_3/ZrO_2 coating. This was followed by YSZ, then the mullite without a bond coat. The mullite coating sprayed over a NiCr bond coat performed very poorly in both endurance trials.

A detailed assessment of the data suggests three potential aspects of heating element degradation based on the thermal and chemical degradation of the coating. Thermal degradation of the coating was noted by observing the power requirements to maintain constant temperature of the heating elements. This is summarized in Figure 12. Here, power level is defined by the voltage (shown as a %heat) to maintain the desired temperature in the final pulse. Those trials requiring higher power levels at the end of the heating schedule to achieve the aim temperature were correlated with reduced life of the ceramic. It was further noted that the lower power trials correlated with those using reduced contact forces, which in turn implied poorer contact between the heating element and substrate. The trials with improved electrode contact may have better heat transfer from the steel into the ceramic, resulting in an increase in power demand and thus coating degradation.

A related degradation may also occur because the heating element may have mechanically bowed or twisted. Figure 13 illustrates a possible orientation of bowing in the electrode as a means of altering the heat conducted to the copper fixture. This could be caused by linear expansion of TZM element constrained in the tooling. The resulting bowing can cause both hot spots due lack of contact with the fixture, as well as surface strains leading to coating fracture.

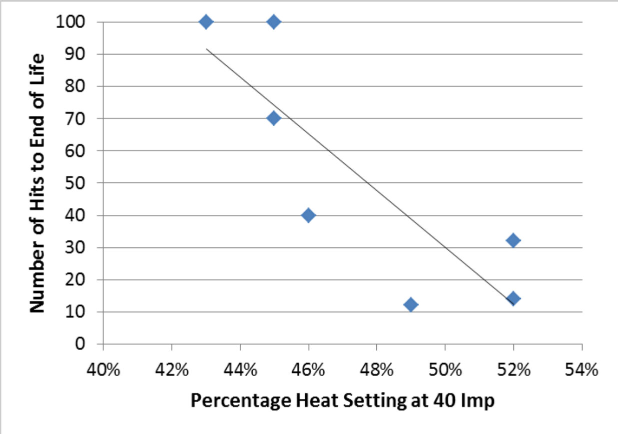

Figure 12. Comparison of Coating Life with Power Level During Testing (%heat shown represents the voltage in the last power pulse in the process)

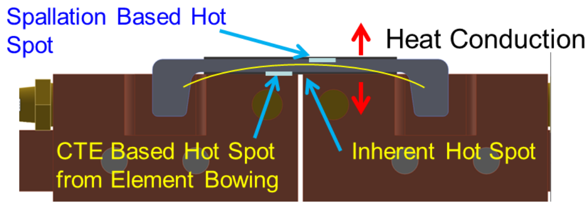

Figure 13. Potential Distortion of the Heating Element during Processing (slight bowing in the electrode may alter the heat conducted to the copper fixture and create a hot spot due to spallation of coating)

Chemical degradation was the result of chemical diffusion and interaction between the substrate and the coating itself. This type of degradation takes several forms; such as a loss of coherency between the bond coat/TZM, or constitutional liquation and related hot cracking.

Spallation was observed primarily as poor coating adherence between the various layers of the deposit. Spalling of the coating may have been caused by several reasons. It is possible that the base layer could interact with the TZM to liquate and cause spalling. Other possible examples are liquation cracking (MoO_3, MoNi, or Cr-based) along grain boundaries in the ceramic. An example of a liquid forming between the ceramic and substrate (here caused by the interlayer reacting with the TZM) is shown in Figure 14. Evidence of how such liquids can penetrate the coating grain boundaries and cause cracking is provided in Figure 15.

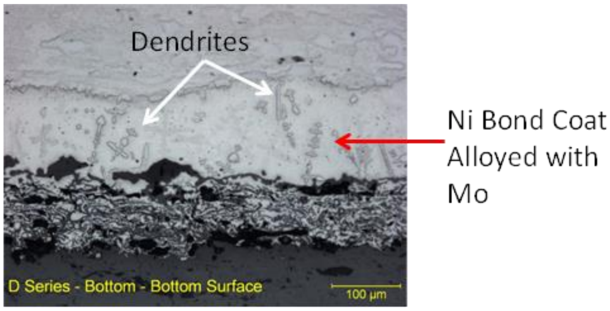

Figure 14. Formation of Liquid at the Interface between the Ni from the Bond Coat and Mo from the TZM

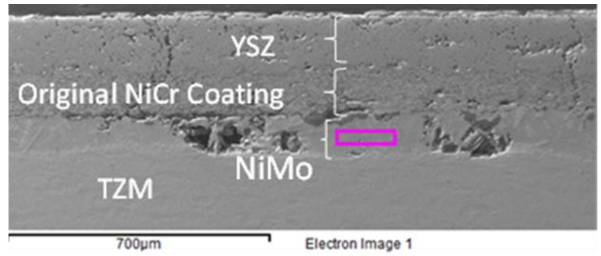

Figure 15. Example of Constitutional Liquation and Subsequent Cracking of the Ceramic Coating

It must be noted here that the degradation of these ceramic coatings has certainly been accelerated by the nature of these endurance tests. The actual IRHT cycles are much shorter, but may also eventually be degraded by the types of issues observed in this study. Still, such accelerated tests provide guidance for the design and selection of future IRHT processes. All of these ceramic systems may provide acceptable performance depending on the temperature regimes and durations defined by the application. However, further improvements to the IRHT system can clearly be obtained by improving the ceramic coating adhesion and toughness, redesigning the heating element shape, and improving the thermal management of the fixture. Based on the present work, implementation of some of the improvements listed below should allow the IRHT process to produce more consistent localized material properties that will reduce material and processing costs in the lightweighting of automotive structures using the IRHT process.

Conclusions

In this study, degradation of ceramic coatings applied to heating elements used for indirect resistance heat treatment (IRHT) of steels was studied. An accelerated durability test was developed, employing a 0.9-mm DP780 steel workpiece. Four types of plasma-sprayed ceramic coatings were produced, namely Al_2O_3/ZrO_2, YSZ, and mullite with and without a bond coat. The heating element substrate was TZM, and the bond coat was a thin layer of APS NiCr. Wear tests were performed up to 100 thermal cycles, composed of 45-s heating and 45-s cooling durations using a specialized IRHT fixture. Failures of the coatings were noted to be related to thermal and chemical degradation effects. Specific conclusions drawn from this work include:

(1) Heating time and temperatures to produce austenitic transformation in steel is related to the C content and the rate of heating. Economic thermal processing needs drive the process to higher temperatures than displayed on the equilibrium phase diagram.

(2) Al_2O_3/ZrO_2 coating provided the best endurance performance overall, followed by YSZ and mullite without a bond coat. All of these ceramic systems may provide acceptable performance if the thermal management of the heating element can be improved (fixture and spallation issues).

(3) Mullite coating with a bond coat promoted gross spallation of the mullite coating and reduced coating durability. This was related to reactions between the bond coat and the substrate TZM, causing constitutional melting.

(4) Improved coating adhesion to the TZM and improved toughness of the coating are needed to improve coating endurance performance. Mechanisms of wear included spalling and fracture of the ceramic itself.

(5) Redesigned element shape or tooling is needed to avoid spallation of coating from the element. Distortion during heating is believed to cause stresses in the element resulting in fracture of the ceramic coating.

(6) Actual IRHT electrode longevity is longer and the accelerated wear results illustrated in this project are integrally related to the long durations per heating cycle. The endurance test used here was designed to test the coating and not be representative of heat treatment cycles planned for future exploitation.

References

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(2) Gould, J.E., Harris, M., Sakkinen, D., and Peterson, W., "Application of direct resistance treatment to localized areas on advanced high strength steel sheet", *Welding in the World,* 2016. DOI 10.1007/s40194-016-0361-5.