Cyclic Oxidation Testing of Aluminized Binary Nickel Chromium Alloys

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Kellogg Honors College Capstone Project

1. Background

High temperature materials are often used in industry in gaseous environments with alternating temperature cycles. An example of such is in turbine blades used in aerospace. The high temperature cycles results in high temperature cyclic oxidation, which degrades the material as it forms oxides [1].

In order to test metals under high temperature cyclic oxidation conditions, researchers often develop testing rigs that move either the sample or furnace in an oscillatory motion to heat and cool the sample. Such tests are often performed in expensive mass-production cyclic furnaces, in thermal gravimetric analysis furnaces (TGAs), or in setups developed by the researchers in their labs.

Coatings produced by a Halide Activated Pack Cementation (HAPC) process help prevent the oxidation and corrosion of metals in high temperature applications. The HAPC process consists of a “pack,” which contains the substrate, the master alloy being added to the substrate (aluminum in this study), a halide activator (sodium fluoride), and an inert filler (aluminum oxide). Packs are then heated in a furnace to allow the master alloy to diffuse into the substrate, resulting in a hard, corrosion-resistant coating [2]. The coating provides a reservoir of master alloy which will react with the environment and protect the substrate.

2. Objective

The objective of this project was to develop an automated horizontal cyclic oxidation setup on a furnace using readily available components. The setup would then be used to test uncoated and coated binary nickel-chromium alloys (with 5 wt.% Cr) at a range of temperatures for 4 cyclic hours (cycles of 1 h in, 15 min out of the furnace).

3. Hypothesis

The aluminized coating will have less mass loss and visible oxidation.

4. Cyclic Oxidation Setup

4a. Design Criteria

The design requirements of the cyclic oxidation setup were in anticipation of future tests to be performed. As such, the criteria were:

-Low cost (excluding cost of furnace and alumina tube) and efficient

-Fully automated to allow for precise cyclic time intervals and to prevent human error

-Horizontal arrangement of the furnace

-Sample placed in an enclosed tube, in which gas may flow through the tube

4b. Design and Development

The main mechanical system was based on a lead screw design powered by a motor. As the motor rotates the screw, the rotational motion is translated into linear motion to guide the furnace-cart assembly on the rails.

The cyclic oxidation setup, shown in Figure 1, can support both open ended or one open, one closed ended tubes by positioning the motor and tube stands accordingly.

The motor, powered with a DC power supply, was controlled with an Arduino microcontroller in conjunction with a relay system. Hall effect sensors were positioned beneath the cart to limit the distance moved by the cart. An LCD screen with buttons provided the user interface (Figure 2). An Arduino program was coded to store user inputs such as cycling time and number of cycles.

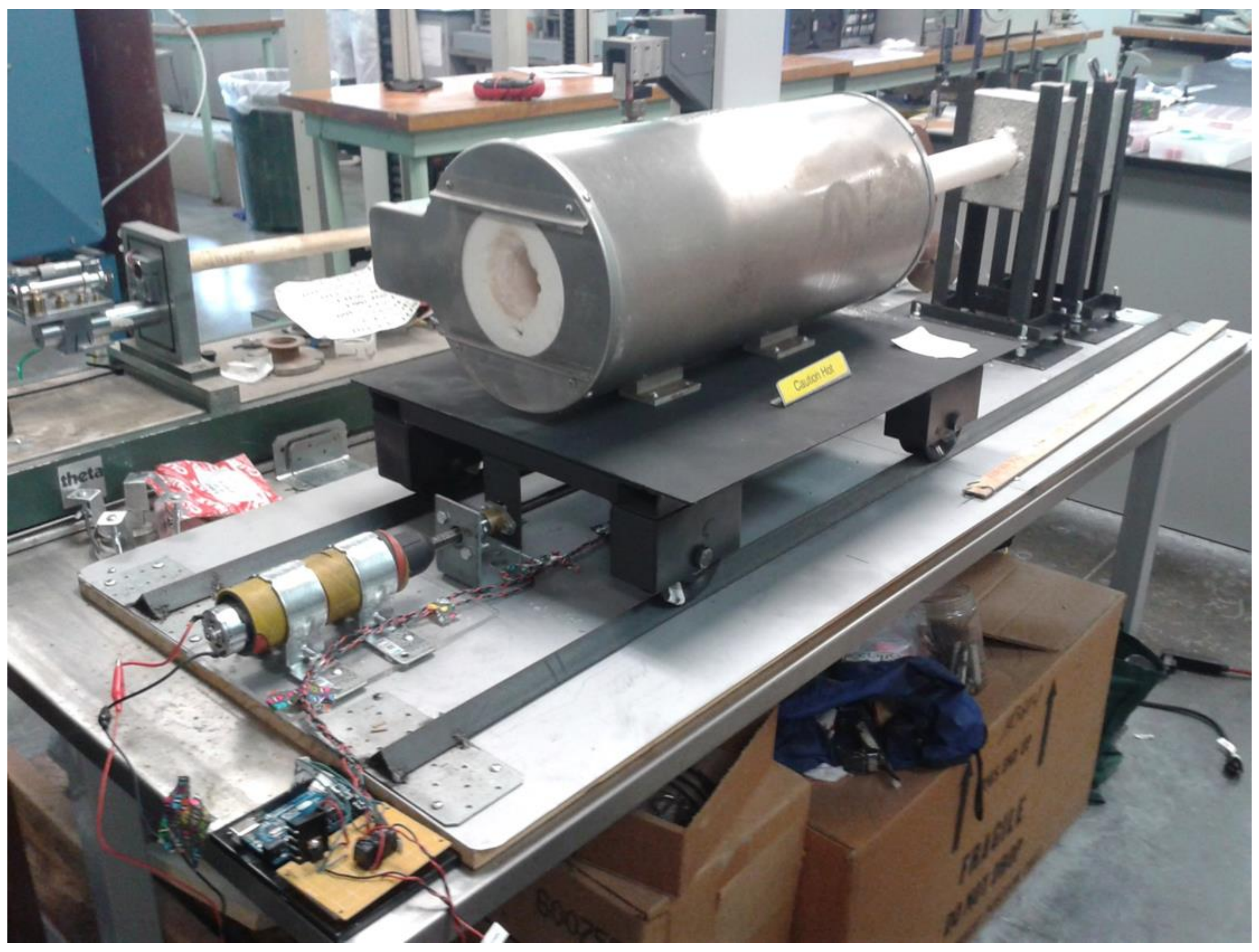

Figure 1: Completed Cyclic Oxidation Setup

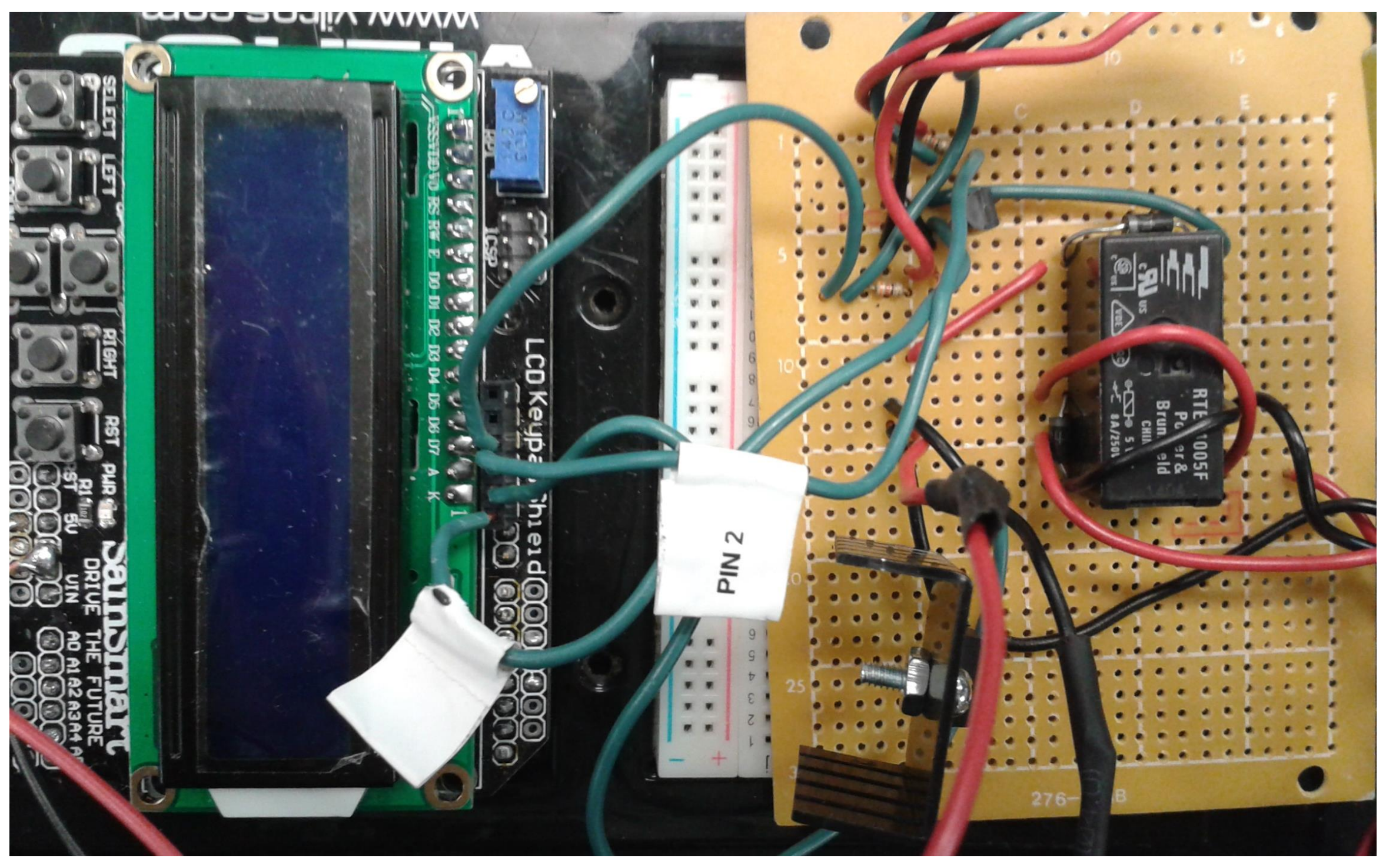

Figure 2: Arduino Microcontroller with LCD Interface Screen and Relay Circuit

5. Results

5a. Macrographs

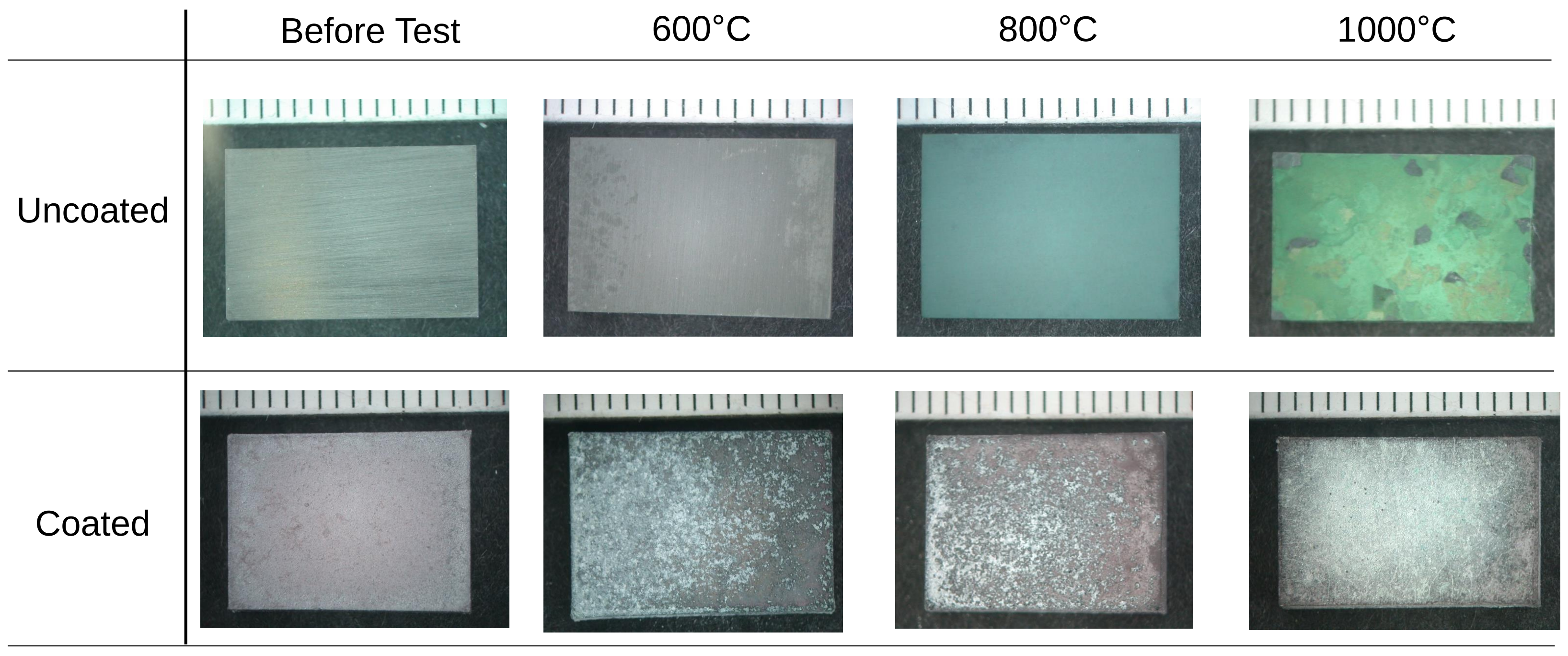

Figure 3: Macrograph images of specimens after 4 h cyclic oxidation test

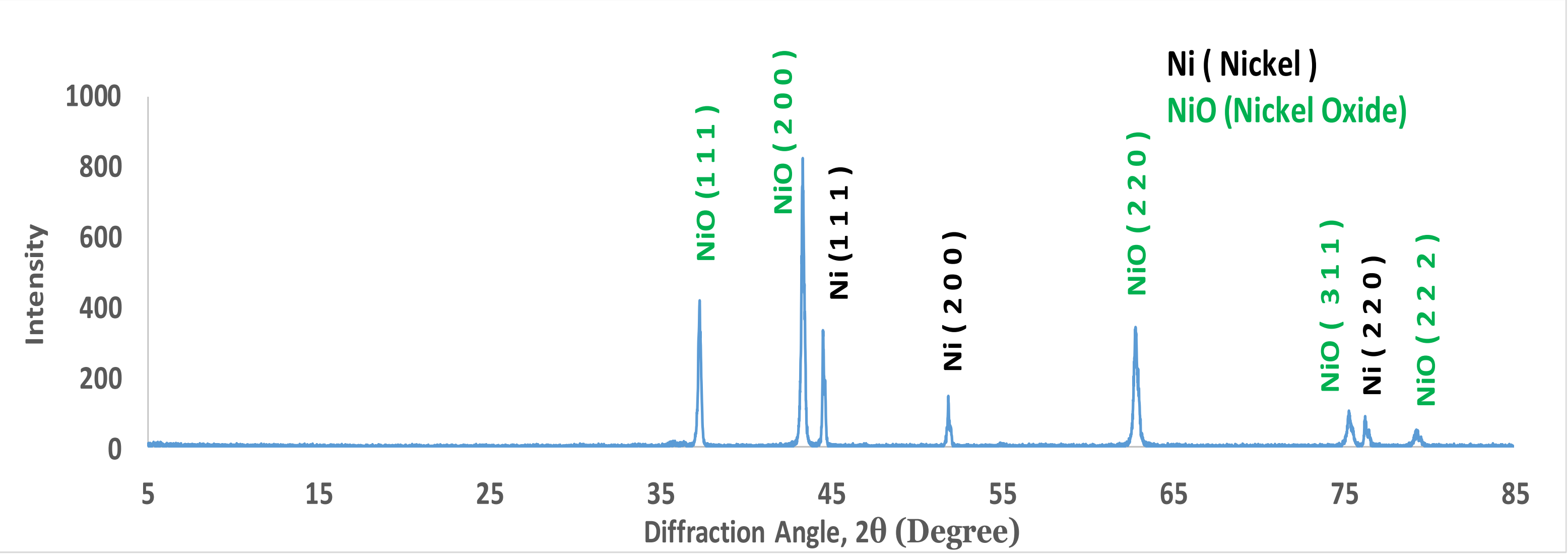

Figure 4: X ray diffraction analysis of an uncoated Ni-5Cr specimen after 4 h cyclic oxidation test at 800°C

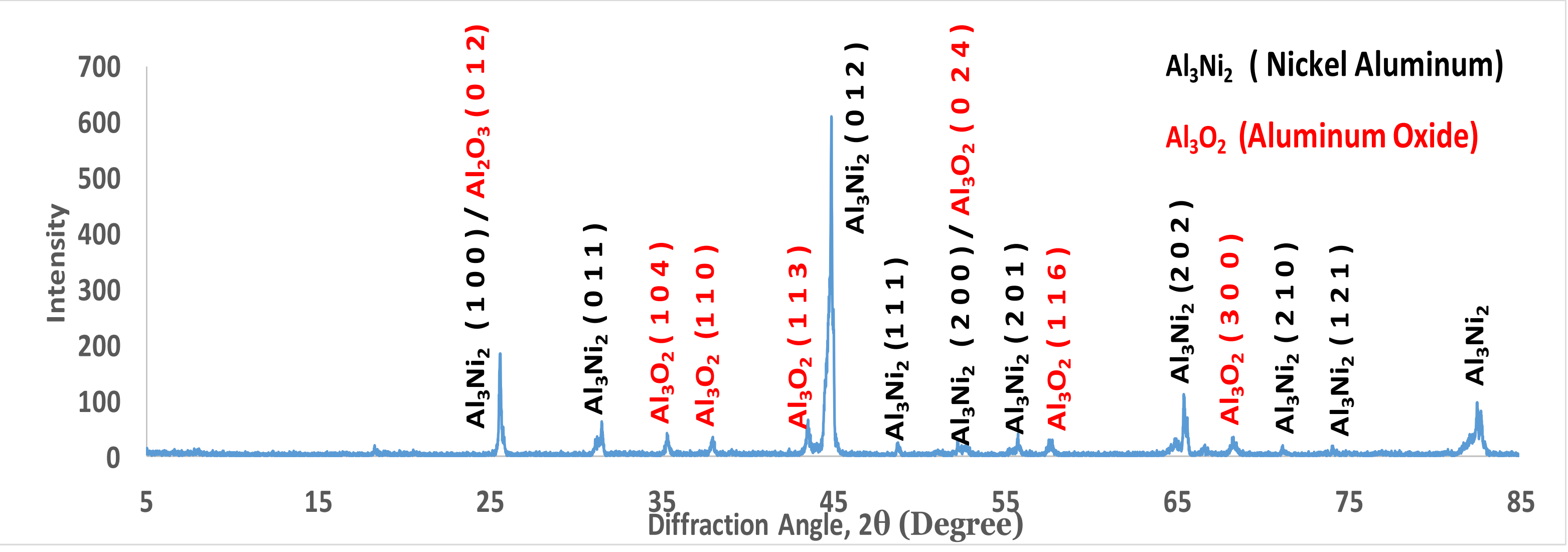

Figure 5: X ray diffraction analysis of a coated Ni-5Cr specimen after 4 h cyclic oxidation test at 800°C

6. Discussion

Visually, the effects of oxidation on the uncoated samples can be seen in the macrographs in Figure 3. For the uncoated samples, as temperature increases, both uncoated and coated samples appear to have more coverage oxide layer. At 600°C. the oxide film partially covered the surface of the uncoated Ni-5Cr sample, while a green-grey oxide formed more extensively on the specimen at 800°C. At 1000°C, the green oxide layer begins to crack and spall off. The spallation is attributed to tensile forces on the surface oxide layer, as a consequence of a larger coefficient of thermal expansion for the oxide [1].

Ni-5Cr specimens aluminized using the HAPC process with NaF as the activator at 950°C for 9 h were also subjected to cyclic oxidation tests. As the temperature increased, the coated specimens exhibited an increase in grey-white specks on the surface.

X ray diffraction (XRD) analysis of cyclic oxidized specimens at 800°C for 4 cycles (each cycle consists of 1 h at temperature and 15 min at ambient) showed a difference in phases detected in the uncoated and coated Ni-5Cr specimens, as shown in Figure 4 and Figure 5. Nickel (Ni) and nickel oxide (NiO) were detected on the surface of the uncoated specimen, while nickel aluminide (Ni_2Al_3) and aluminum oxide (Al_2O_3) were detected on the surface of the coated specimen. The Ni_2Al_3 phase is attributed to the diffusional coating. The grey-white specks were most likely the aluminum oxide formed on the surface during oxidation. It is to be noted that no chromium oxides were detected on either oxidized samples.

The mass per unit surface area of the coated samples generally increased with increasing in temperature, due to the formation of oxides on the surface of the specimens. At 600°C and 800°C, slight mass increases of 0.001 and 0.012 mg/cm^2 on uncoated samples, respectively, were observed. At 1000°C, the uncoated specimen had a mass change of –0.267 mg/cm^2 due to the loss of material during spallation. The coated specimens had mass increases similar to those of the uncoated specimens at 600°C and 800°C, but differed greatly at 1000°C with 0.216 mg/cm^2.

7. Conclusions and Future Work

An operational cyclic oxidation setup was built, and testing on uncoated and coated binary nickel-chromium alloys was performed for 4 cycles with temperatures ranging from 600°C to 1000°C.

Future analysis using optical microscopy and scanning electron microscopy will be used to compare the effect of chromium within the coating layers before and after the cyclic oxidation testing. Additional cyclic testing will be performed with longer testing durations to further characterize the growth of the oxides with change in time.

8. Acknowledgements

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10. References

1. Barrett, C. A., and C. E. Lowell. "High temperature cyclic oxidation furnace testing at NASA Lewis Research Center." (1981).

2. V. A. Ravi, “Pack Cementation Coatings,” in *Corrosion: Fundamentals, Testing and Protection*, Vol. 13A, ASM Handbook, ASM International, (Materials Park, OH: ASM International), (2003): pp. 763-771.