THE STUDY OF SURFACE PHASES ON ALUMINIDE COATINGS USING X-RAY DIFFRACTION

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1. Background
Aluminide coatings are widely used to protect materials of construction subject to harsh operating conditions such as high temperatures and corrosive environments. Halide activated pack cementation (HAPC) is an economical way to produce aluminide coatings that enable materials to withstand a more corrosive environment [1]. Aluminide coating typically consisted of intermetallic compound. Intermetallic, such as AlFe, can form in the coating after exposure to a corrosive environment and cause significant reduction in the performance of the coating [2].

The transformation of surface phases depends on the aluminide coating.

2. Objective
To investigate the effect of activator and temperature on transformation of surface phases of the aluminide coating.

3. Hypothesis
The transformation of surface phases depends on coating temperature and the thermodynamic stability of activators.

4. Materials & Methods
3.1 Preparation of the pack
UNS S30400 austenitic stainless steel samples were metallographically prepared to a 600 grit finish. Mass and dimensions of the sample were recorded. The samples were then degreased and cleaned by ultrasonic cleaning in methanol, followed by cleaning with soap and a demineralized water rinse. A mix of aluminum, aluminum oxide, and an activator (NH₄Cl, AlCl₃, NaCl, NaF, AlF₃, Na₃AlF₆) was prepared. The mixture was thoroughly mixed, then blended in a roller mill. The blended pack and the sample were then placed into an alumina crucible and sealed using ceramic cement. After a curing process, the pack was placed in a furnace and heated to temperatures of 650-850 °C for 9 hours. After the furnace cooled down, the pack was extracted from the furnace and opened to remove the sample. The sample was then thoroughly cleaned, dimensioned, and analyzed using X-ray diffraction.

3.2 Characterization of coatings
The surface phases were identified by X-ray diffraction using a Cu Ka (λ = 1.540560 Å) radiation source, 40 kV tube voltage and the beam current of 44 mA. Data were collected by step scanning from 5° - 85° with step size of 0.02° (2θ).

5. Result

The results indicate that process temperatures and the thermodynamic stability of the activator affect both the type and composition of the surface phases. In general, the aluminide coatings exhibit multiple phases at the lowest coating temperature (650°C) and transform into a single phase at the higher coating temperature (850°C). Figure 1 maps out the different phases as a function of coating temperatures and activators. Surface phases produced using NH₄Cl undergo the most number of transformations, in regard to the dominant phase, the temperature range studied (Figure 2). At 650°C, the NH₄Cl-activated pack produces three phases with AlFe₂ as the most prominent phase (57.4 wt%) co-existing with Al₅FeNi (38.6 wt%) and AlFe₃ (4.0 wt%). When the coating temperature was increased to 750°C, these multiple phases evolved into an aluminum rich phase (Al₅FeNi) that transformed into Al₂Fe₂ at 850°C. Similar phase transformation behavior in NaCl and Na₃AlF₆-activated packs were observed. At 650°C, Al₂Fe₂ (97.9 wt%) and AlFe (2.1 wt%) were formed. When the coating temperature increased to 750°C and 850°C, AlFe remained as the most abundant phase. Increased amount of the AlFe intermetallic was observed with increases in coating temperature. This is supported by the observation of higher peak intensities for the AlFe phase (Figure 3). The nature of the phase transformation in NH₄Cl, NaCl, and Na₃AlF₆-activated packs could be further understood by examining the thermodynamic stability of these salts (Figure 4). NH₄Cl is the least stable activator when compared to NaCl and Na₃AlF₆. Therefore, the stability of activator plays an important role in the nature and amount of the surface phases. The most stable activator maintains a stable pack atmosphere thereby resulting in more gradual changes at the surface. The unstable activator, on the other hand, is likely to create changes in the pack atmosphere, thereby resulting in more changes at the surface.

6. Discussion

The surface phases of aluminized UNS S30400 were successfully quantified using XRD. The whole pattern fitting method revealed that Al₅Fe₂ is the most common phase for all process parameters utilized in this study. Al₂Fe₂ and AlFe were observed to co-exist when NaCl, NaF, and Na₃AlF₆ activators were used. The thermodynamic stability of the activators was found to have an impact on the prominent phase of the coating, e.g., the prominent phase formed using the least stable activator (NH₄Cl), is Al₂Fe₂ at 650°C, Al₅FeNi at 750°C and Al₂Fe₂ at 850°C. However, for the most stable activator (Na₃AlF₆), the prominent phase is AlFe for all coating temperatures.

7. Summary and Conclusions

The surface phases of aluminized UNS S30400 are sensitive to the activator and temperature. The X-ray diffraction analysis indicates that the surface phases are transformed at different rates depending on the activator and temperature.

8. Future Work
XRD analysis will be used to further investigate the following areas:
- Effect of surface roughness on XRD pattern generation
- Crystallographic size determination
- Residual stress determination

9. References

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