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# Oxidation Kinetics of Nicrofer-6025HT for Use in Elevated Temperature Electrochemical Devices

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# Background

Recent advances in high temperature electrochemical devices have prompted research into potential materials for component fabrication. The manifold must house and be bonded to solid state mixed ionic and electronic conductor (MIEC) components.

Devices include: oxygen separation units, solid oxide fuel cells, and carbon capture and sequestration systems.

Devices, using the current design, require a manifold material that meets the following requirements:

- Maintain structural stability at elevated temperatures. Nickel alloys are strong candidates.
- A CTE that closely matches that of the ceramic components.
- Produces an oxide surface that allows reactive air brazing (RAB) between dissimilar materials.
- Produces a discontinuous internal oxide layer for devices that require electrical conductivity.
- Forms a continuous (non-spalling) outer oxide layer that enables the formation of a hermetic RAB seal during brazing.

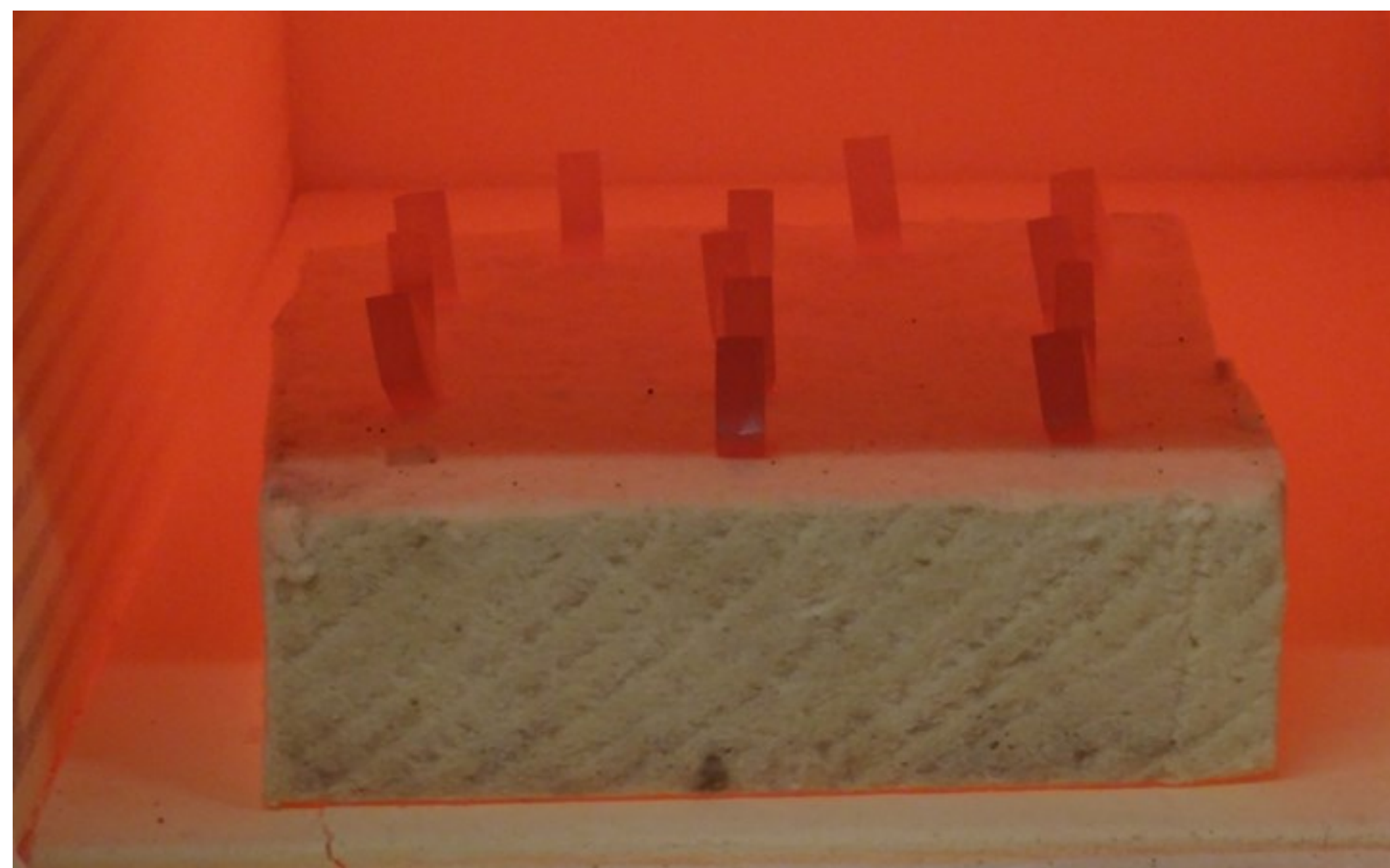


Fig. 1: Photo of samples being oxidized in furnace.

**Nicrofer-6025HT** — Produced by ThyssenKrupp VDM, is a nickel based alloy that is being investigated for use in MIEC membranes.

It is predicted that Nicrofer oxidation will follow the parabolic growth law:

$$\left(\frac{\Delta m}{A}\right)^2 = k_p t$$

where  $\Delta m$  is the change in mass,  $A$  is the surface area of the specimen,  $k_p$  is the parabolic rate constant, and  $t$  is time.

Nicrofer is expected to follow Type II oxidation, having high Cr (> 15%) and low Al (1% < Al% < 3%).<sup>[1]</sup> It is predicted that the oxide scale will consist of a  $\text{Cr}_2\text{O}_3$  outer scale with  $\text{Al}_2\text{O}_3$  internal oxides.

## OXIDATION KINETICS OF NicroFER-6025HT FOR USE IN ELEVATED TEMPERATURE ELECTROCHEMICAL DEVICES

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### Procedure

Samples were oxidized at three temperatures for designated times (Table 2). The weight was measured before and after oxidation to determine the change in mass.

Table 1: Test Matrix for Oxidation Studies

Time (days)	Hold Temperature		
	800 °C	850 °C	900 °C
0.25	X	X	X
1	X	X	X
2	X	X	X
5	X	X	X M
10	X (2)	X (2)	X (2) M
15	X	X	X
20	X	X M	X M
25	X (2)	X (2)	X (2)
30	X	X	X
40	X	X	X M
50	X (2)	X (2)	X (2)

X = Furnace Samples (2) = Duplicate Samples  
M = SEM Microstructural Analysis

SEM-EDS was performed on preselected samples to measure the oxide thickness and to perform a basic characterization of the oxide scale.

### Results

A parabolic curve fit was applied to the data set to establish a relationship between the relative weight gain and the time of oxidation (Fig. 1 & 2).

Parabolic fits at 850 °C and 900 °C imply that the parabolic growth law represents the oxidation mechanism of Nicrofer at these temperatures.

SEM analysis (Fig. 4) verified Type II oxidation with an outer Cr-rich oxide and acicular Al-rich oxides penetrating into the nickel alloy matrix.

Oxide thickness were measured in select SEM images for comparison with weight gain values (Table 2).

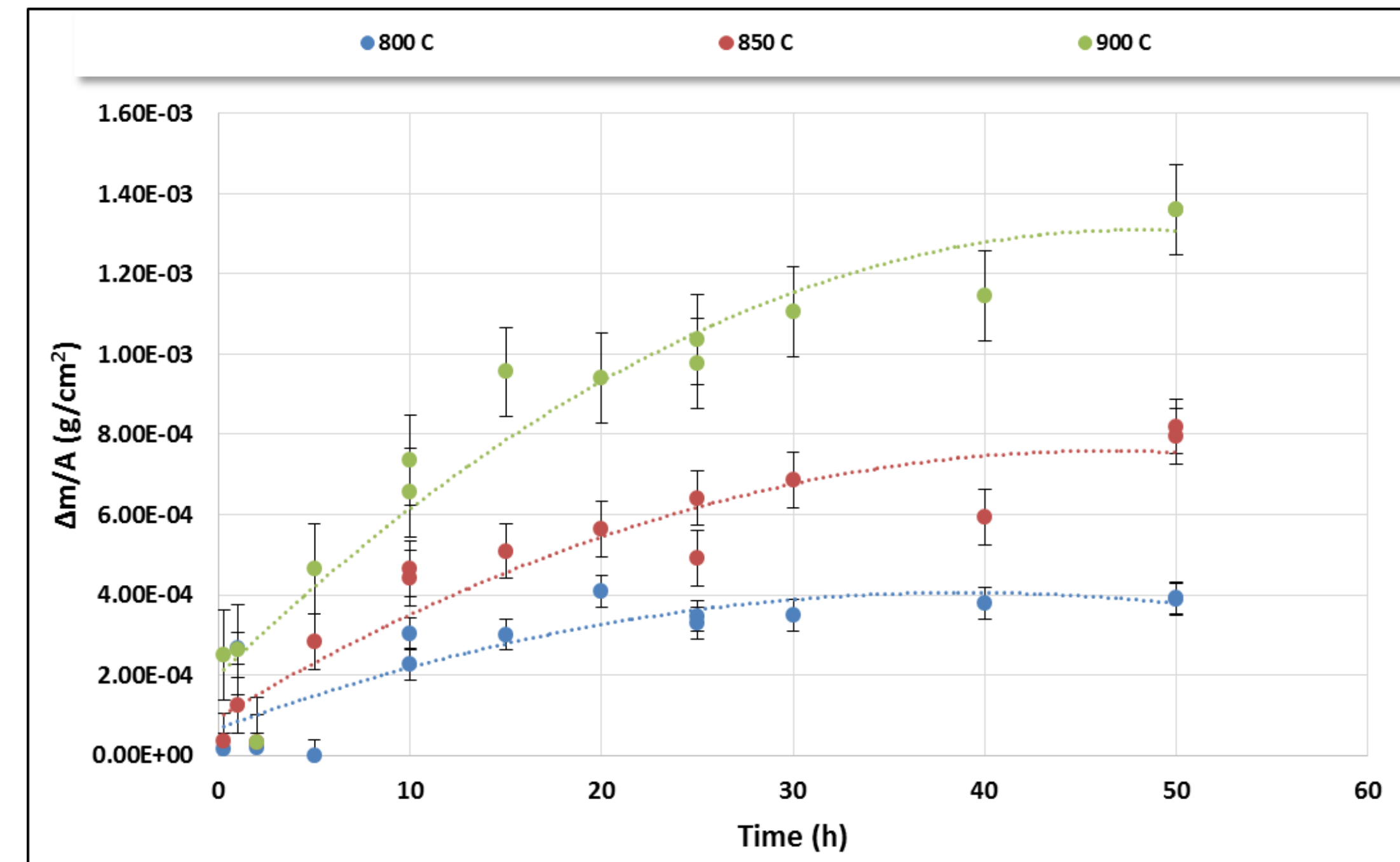


Fig. 2: Relative weight gain as a function of time at the oxidation temperature.

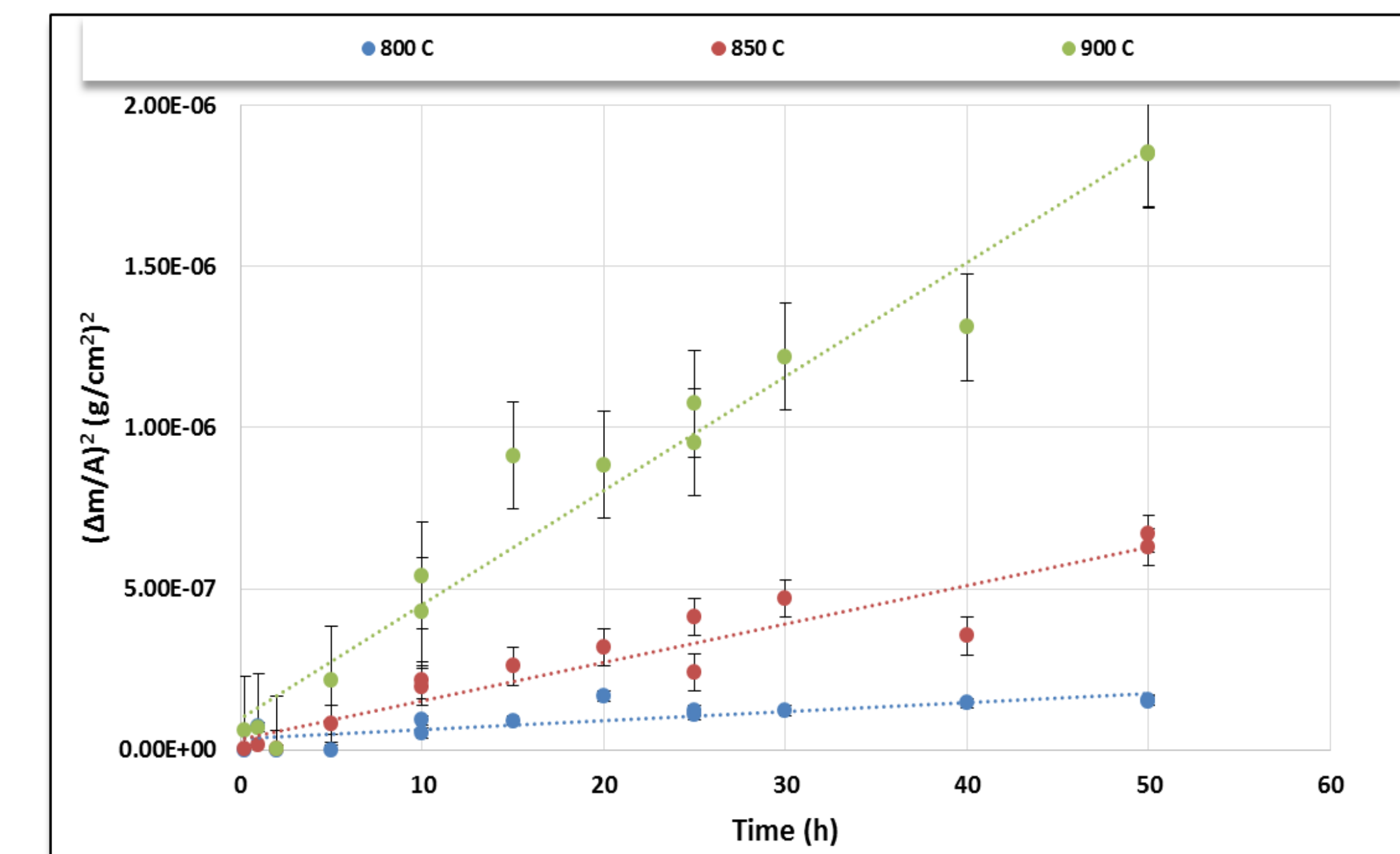


Fig. 3: Relative weight gain squared as a function of time at the oxidation temperature.

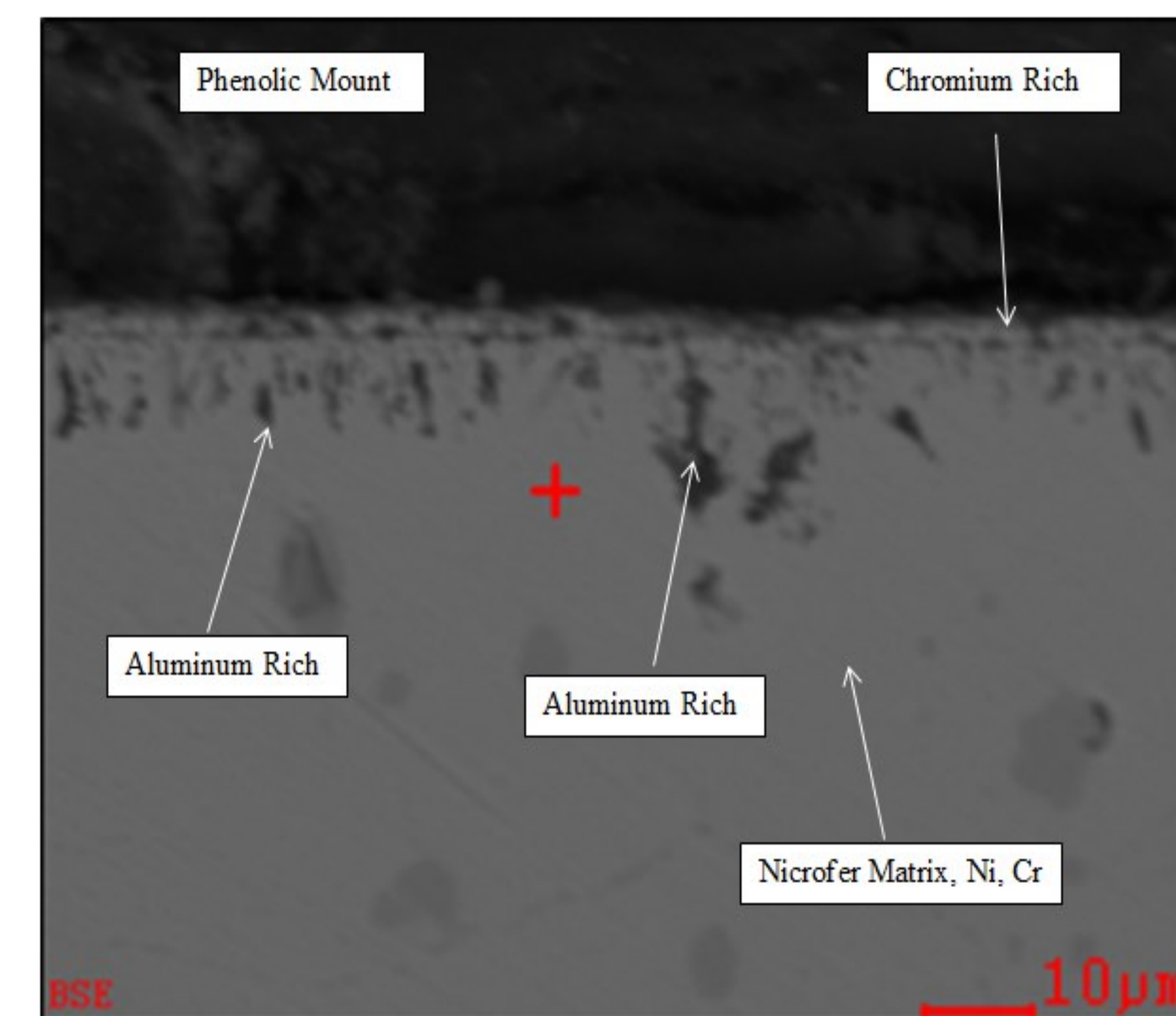


Fig. 4: Typical oxide layer microstructure (850 °C for 20 days).

Table 2: Oxide Thickness Measurements

Temperature (°C)	Time (days)	Average Internal Oxide Penetration Depth (μm)	Average External Oxide Thickness (μm)	Average Total Oxide Product Zone Depth (μm)
900	5	9.3	1.3	10.6
900	10	11.4	1.5	12.9
850	20	11.0	1.8	12.8
900	20	16.8	2.5	19.3
800	50	17.4	1.5	18.9

### Discussion

Comparison was made between the rate constants determined in this research with those determined by other researchers (Table 3). The rate constants differed by one to two orders of magnitude.

The work by Pfeifer<sup>[2]</sup> consisted of determining the rate constant of samples with a 1200 grit SiC polished surface roughness. Parabolic rate constants were also developed by Yang et al.<sup>[1]</sup> at PNNL. The surface condition of these samples is unknown.

Several possibilities exist concerning the discrepancy in rate constants. Parabolic rate is dependent on alloying composition, thermal history and surface conditions.<sup>[1]</sup> The same alloy was used in all research. The surface roughness differed in the Pfeifer research, however the thermal history is unknown. The thermal history and surface conditions are unknown in the Yang et al. research.

Table 3: Comparison of parabolic thickening rate ( $k_p$ ) results.

Temperature (K)	Current Study	Pfeifer <sup>[3]</sup>	Yang et al. <sup>[2]</sup>
	$k_p$ ( $\text{g}^2/\text{cm}^4 \text{ s}$ )	$k_p$ ( $\text{g}^2/\text{cm}^4 \text{ s}$ )	$k_p$ ( $\text{g}^2/\text{cm}^4 \text{ s}$ )
800	7.81E-13	N/A	2.2E-15
850	3.31E-12	1E-14	N/A
900	9.81E-12	1E-13	N/A

### Conclusions

The Nicrofer oxidation exhibited parabolic growth mechanism at 850 and 900 °C indicating that the reaction was primarily diffusion controlled. At 800 °C, a more complicated oxidation mechanism was observed that was neither linear nor parabolic.

SEM-EDS analysis verified Type II oxidation behavior with a Cr-rich outer layer and an Al-rich internal region as predicted based on the alloy composition.

The thickening kinetics analyzed in the current study were significantly more rapid than in the previous studies by Pfeifer<sup>[2]</sup> and Yang<sup>[1]</sup>.

#### References

- [1] Yang et al., Selection and Evaluation of Heat-Resistant Alloys for SOFC Interconnect Applications, Journal of Electrochemical Society (2003)
- [2] Pfeifer, BS Thesis, Alfred University (2010)

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