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Exfoliation Propensity of Oxide Scale in Heat Exchangers Used for Supercritical CO_2 Power Cycles

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Abstract

Supercritical CO_2 (sCO_2) Brayton cycle systems offer the possibility of improved efficiency in future fossil energy power generation plants operating at temperatures of 650°C and above. As there are few data on the oxidation/corrosion behavior of structural alloys in sCO_2 at these temperatures, modeling to predict the propensity for oxide exfoliation is not well developed, thus hindering materials selection for these novel cycles. The ultimate goal of this effort is to provide needed data on scale exfoliation behavior in sCO_2 for confident alloy selection.

To date, a model developed by ORNL and EPRI for the exfoliation of oxide scales formed on boiler tubes in high-temperature, high-pressure steam has proven useful for managing exfoliation in conventional steam plants. A major input provided by the model is the ability to predict the likelihood of scale failure and loss based on understanding of the evolution of the oxide morphologies and the conditions that result in susceptibility to exfoliation.

This paper describes initial steps taken to extend the existing model for exfoliation of steam-side oxide scales to sCO_2 conditions. The main differences between high-temperature, high-pressure steam and sCO_2 that impact the model involve (i) significant geometrical differences in the heat exchangers, ranging from standard pressurized tubes seen typically in steam-producing boilers to designs for sCO_2 that employ variously-curved thin walls to create shaped flow paths for extended heat transfer area and small channel cross-sections to promote thermal convection and support pressure loads; (ii) changed operating characteristics with sCO_2 due to the differences in physical and thermal properties compared to steam; and (iii) possible modification of the scale morphologies, hence properties that influence exfoliation behavior, due to reaction with carbon species from sCO_2. The numerical simulations conducted were based on an assumed sCO_2 operating schedule and several generic heat exchanger channel shapes and cross-sectional areas. Implications for the evolution of stresses in the oxide scales formed on sCO_2 heat exchangers, and ensuing critical oxide thicknesses for exfoliation, were derived and compared with expectations for an equivalent conventional tubular heat exchanger in a steam cycle (for a given alloy).

Introduction

Power generation facilities based on a Brayton-cycle using sCO_2 have the potential to produce low cost electricity due to their reduced complexity and smaller component sizes than those for steam-based Rankine cycles, in which the steam expansion to low pressures results in large turbine/condenser components [1, 2]. However, proposed open and closed Brayton cycle systems employing sCO_2 as the working fluid present challenging requirements of strength and environmental resistance for the materials specified for the hot flow-path components [1]. These components would operate at high pressures (200-300 bar) and/or high temperatures (500°-700°C for closed-cycles and up to 1150°C for open-cycles). A major component in these sCO_2 cycles is the recuperator, a heat exchanger with two streams of sCO_2, one entering at high temperature but low pressure (turbine exhaust) and another entering at low temperature but high pressure, which is pre-heating the fluid entering an intermediate heat exchanger in a closed cycle, or a combustor in the case of oxy-fueled systems.

This study addresses the materials aspects related to the oxidation/corrosion behavior of structural alloys in sCO_2 as applied to the recuperator, aiming to highlight modeling tools to predict the relevant issues of oxide scale growth, failure, and exfoliation, in order to aid the materials selection process for these novel cycles.

Models for exfoliation propensity

For steam boiler tubes, the initial approach to scale failure and exfoliation was visualized by superimposing the evolution of strain in the scales on the exfoliation diagrams ('Armitt Diagrams') developed by Manning et al. of the Central Electricity Research Laboratory (UK) [3-6, 8]. The driving force for scale exfoliation is the accumulation of strains in oxide scales that result from their growth under the physical constraints of the flow-path configurations, and excursions in heat flux, steam temperature and pressure, and flue gas temperature associated with normal boiler operation [3-7]. Scale failure and exfoliation are mainly triggered by temperature variations experienced by the steam boiler, particularly during shutdown and start-up events. These modified 'Armitt diagrams' are essentially maps of various scale failure criteria, including cracking and exfoliation, appropriate for the system considered. While this information can be used to assess the general effect of operating parameters on the possibility of scale damage and exfoliation, the diagrams cannot indicate the extent of the exfoliation problem, e.g., the mass of oxide exfoliated and propensity for blockage. Thus, only qualitative predictions of exfoliation events are possible using these diagrams.

An approach aimed at quantifying exfoliation, which received much less attention than the Armitt diagram, was presented in the same report from 1978 [4], in which a "fraction of exfoliated area" concept was introduced. In the macro photograph of a longitudinal steam tube cross-section shown in Figure 1a, an area of exfoliated oxide scale is shown at the top of the picture. Based on experimental data, such as the one shown in Figure 1a, the circumferential area fraction of scale exfoliated at each location along the flow channel was found to depend on of the elastic strain energy accumulated in the scale at that location as indicated in Figure 1b. By considering this "fraction of exfoliated area" concept, an exfoliation model was developed [8] by incorporating the effect of oxide regrowth after exfoliation events.

(a)

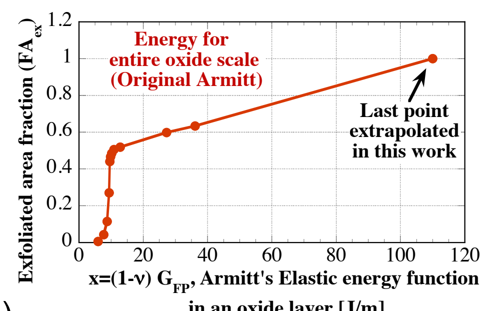

(b)


Figure 1. (a) Example of area of exfoliated scale from a steam boiler tube and (b) fraction of exfoliated area as a function of stored elastic energy function [3, 4].

A flow chart is shown in Table 1 for the exfoliation and blockage model described in this Section. By calculating the state of strain and stress and ensuing elastic strain energy distribution, the "fraction of exfoliated area" and the volume of exfoliated scale can be calculated at each location along the flow-path. Then, the total volume of exfoliated scale can be calculated for the entire heat exchanger loop simply by summing the volumes that were exfoliated at all locations along the tube in the loop. A blockage model then was developed [9] by post processing the total volume of exfoliated scale to obtain the area fraction that would be blocked by scale deposits in tube bends using assumptions of the geometry and size of the deposit(s).

Table 1. Flow-chart of computational models for predicting area fraction of flow cross-section blocked by scale deposits illustrating the input, model or analysis involved, and output.

Step	*Input*	*Model/Analysis*	*Output*
1	Operational T(t) and P(t), Shut-down cycles Oxide kinetics Geometry of flow-path	Stress analysis	State of strain/stress
2	Strain/stress	Algebraic	Elastic strain energy
3	Elastic strain energy	Armitt's elastic energy function	Fraction of exfoliated scale area
4	Fraction of exfoliated area Oxide thickness Length of flow-path	Algebraic	Total volume of exfoliated scale
5	Flow-path shape including turns Location of deposit sites	Geometrical	Geometry of deposit sites Number of deposit sites
6	Total volume of exfoliated scale, Geometry/size of deposit Number of deposit sites	Geometrical	Blockage area (area fraction of flow cross-section blocked by scale deposits)

To illustrate the capability of the exfoliation and blockage model to assess exfoliation propensity as a function of alloy material, operational conditions in a steam boiler (pressure, temperature, and shut-down events), an example is briefly presented in this section for 22.25 mm OD superheater tubes made of TP347H and its fine-grained variant TP347HFG. The tube length was 19m and its thickness changed from 7.75 to 9.1 mm at 11 m. More detailed information on the temperature and oxide thickness distribution along the tube and other operational conditions can be found in [9]. The ratio of deposition length to the tube ID was considered to be 5, and boiler-shut down events were assumed to occur at 6-month intervals. The results were generated as a function of the steam outlet temperature (steam/metal temperature measured in the boiler penthouse). The predicted mass of magnetite exfoliated from the entire tube is shown in Figure 2 for the TP347H and TP347HFG tubes. Overall, it was found that the mass of exfoliated magnetite increased with increasing steam temperature (hence rate of oxide growth), as anticipated.

The results for the amount of scale exfoliated were converted to predictions of the percentage of blocked cross-sectional area of the tube for a horizontal scale deposit that is 5 x tube ID long. The results for the calculated blocked cross-sectional area of the tube are shown in Figure 3, and indicate that the blockage fraction is very high for the first three shut-down events for the TP347H tube, dropping to levels below the 50% acceptable threshold for subsequent outages. This result was found to be in agreement with plant experience [9]. For the TP347HFG tube, the blockage fraction was found to exhibit significantly lower values than for the TP347H tube, with the highest blockage fraction occurring during the fourth and fifth shut-down events.

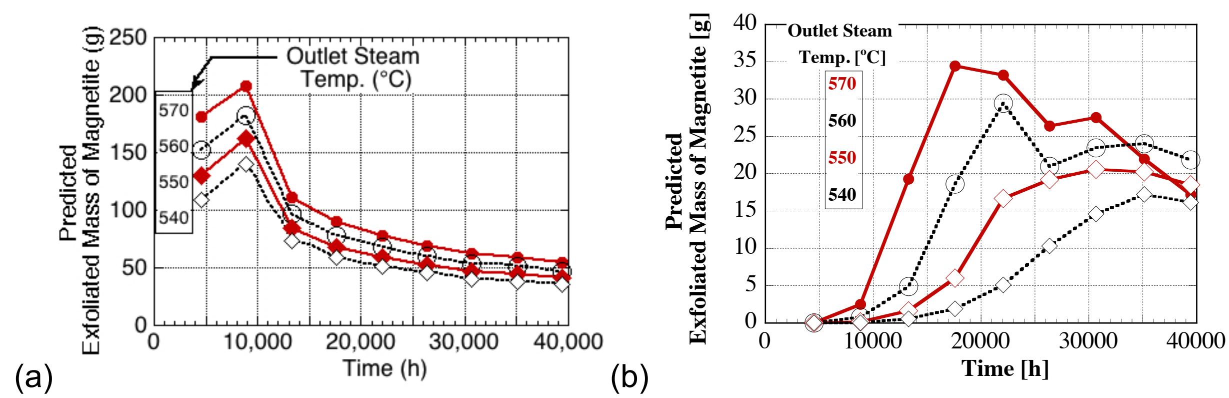


Figure 2. Total mass of magnetite predicted to exfoliate from the whole length of a single superheater tube of (a) TP347H and (b) TP347HFG as a function of time and outlet steam temperature.

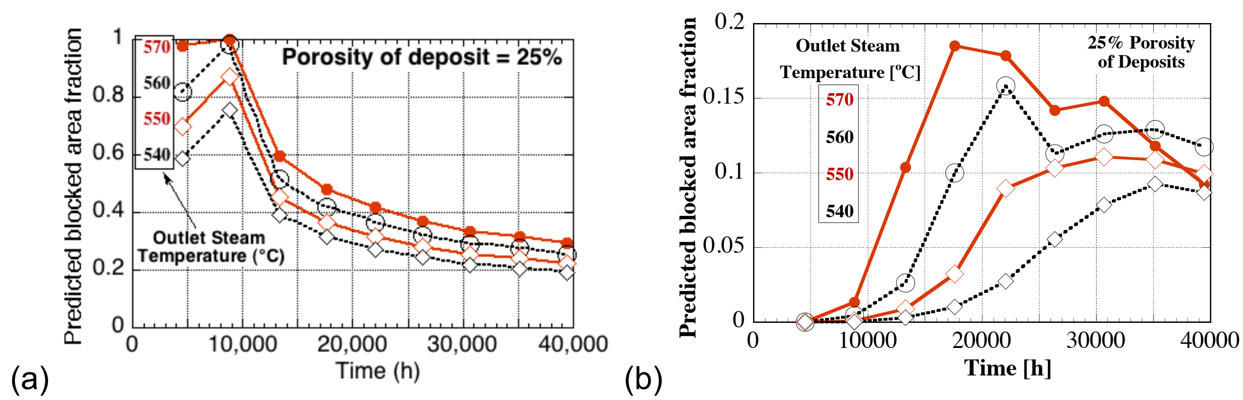
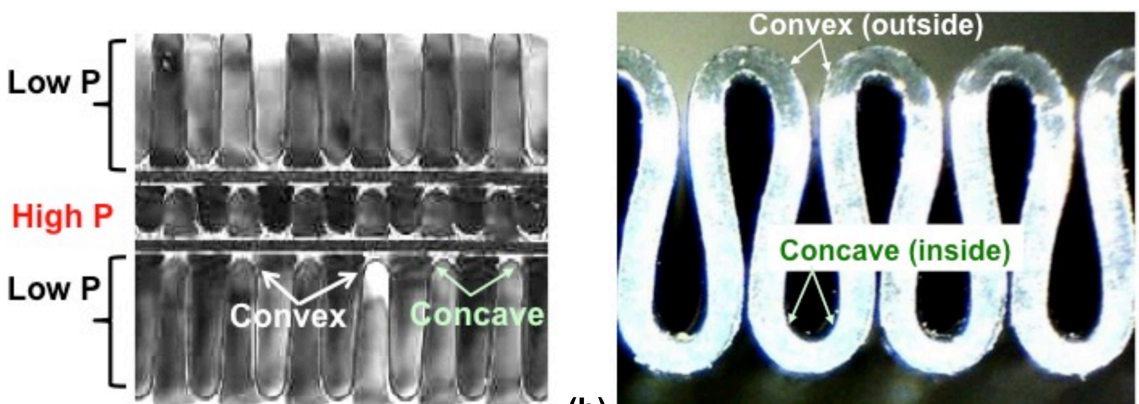


Figure 3. Predicted fraction of tube flow section blocked due to magnetite exfoliation from a single superheater tube of (a) TP347H and (b) TP347HFG as a function of time and outlet steam temperature.

Flow-path geometry for sCO_2 heat exchanger and superheater in a steam boiler

Generic data on design of recuperators intended for use in the sCO_2 Brayton cycle were provided by Brayton Energy, Inc. A current concept for such a recuperator is illustrated in Figure 4. The sCO_2 channels for (i) the high-pressure side, and (ii) the low-pressure side are bundled together in layers (Figure 4a), creating pressure and temperature gradients across the enclosure boundaries between the two main flow channel bundles. The cross-sectional geometries for the flow passages indicate that the alloy surfaces exposed to sCO_2 involve both convex and concave shapes. It has to be mentioned that the maximum allowable thickness of the total oxide scale is limited by the metal thickness and hydraulic diameter of the channels. In small cross-sectional channels, e.g., with hydraulic diameters of 200 to 500 μm, the thickness of adherent oxide scales alone may be sufficient to choke the flow, or significantly reduce the heat transfer performance due to the small thermal conductivity of the oxide scale. Using the oxide growth kinetics for the alloy in question, the time taken at a given temperature for the oxide thickness to reach its maximum allowable value can be calculated. In Figure 5, data on isothermal growth of oxide scales for alloys T91 and TP347H are shown. The results shown in Figure 5a for T91 alloy indicate that the total oxide scale thickness would reach a critical value (for a metal wall thickness of 200 μm) in 16,000h; 3,650h; and 966h at operating temperatures of 600, 650, and 700^{o}C, respectively. The same critical thickness would be reached for alloy TP347H in 4,660h and 1,500h at operating temperatures of 650 and 700^{o}C, respectively. Thus, oxide thickness alone may be enough to cause blockage of flow channels with small geometries, or significantly reduce the performance after only limited service time.



(a) (b)

Figure 4. Cross-sections of a heat exchanger: (a) low-pressure and high-pressure flow paths assembly, and (b) detail on fold geometry used to make each flow-path. Metal thickness is 0.2 mm.

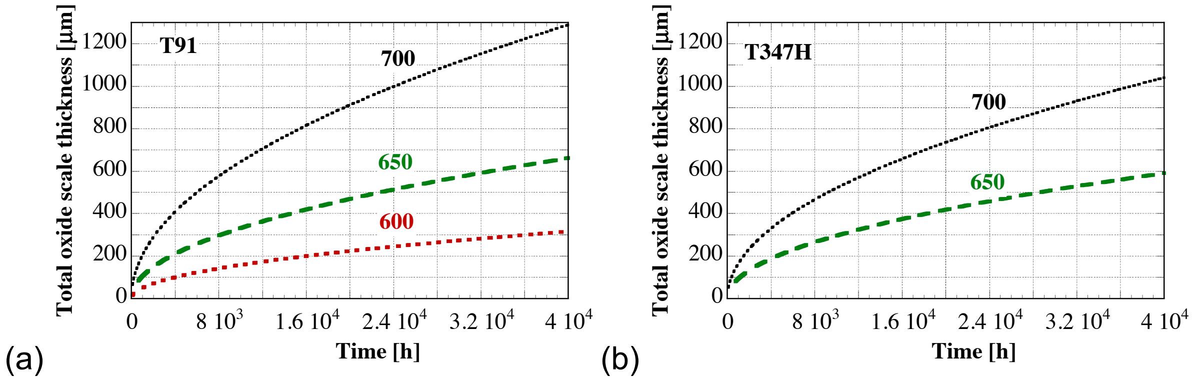


Figure 5. Oxide growth in absence of exfoliation at isothermal conditions for (a) TP91 at 600, 650, and 700 °C and (b) T347H at 650 and 700 °C.

Generic data associated with these geometrical features are summarized in Table 2, together with a side-by-side comparison with superheater tubes in a steam boiler, and include: the number of bends in the longitudinal flow-path; location of possible blockage; and shapes of the blocking deposits. Since some of these geometrical data will critically affect quantitative prediction of the extent of blockage following scale exfoliation, the values shown here should be considered to be preliminary.

Table 2. Typical dimensions and geometries of flow paths for sCO_2 recuperators as compared to those in a superheater steam boiler tube.

Fluid/Size/Property	*sCO_2 Channels*	*Steam Tubes*
Channel shape	Variable curvature	Circular
Oxide growth location	Inside channel (concave) Outside channel (convex)	Inside channel (concave)
Metal thickness [mm]	0.2	7 to 10
Internal hydraulic diameter [mm]	*0.2 to 0.4	20 to 30
Channel length [mm]	500 to 1,000	20,000 to 25,000
Ratio channel length to ID radius	1,200 to 5,000	600 to 1,200
Number of 90° or 180° bends per channel	**None	1
Location of possible blockage	Tube exit	Bends
Shape of blockage	Horizontal, or in the bend at channel exit	Horizontal, or inclined in bends

*ID radius of curvature 0.2 to 0.6 mm

**Bend at channel exit in the current design

Aside from the geometrical differences between the flow paths of the sCO_2 and steam heat exchangers, differences in thermodynamic properties of these two fluids also are important considerations for exfoliation modeling. The thermodynamic and fluid properties used to date in this model, as well as heat exchanger geometries and overall plant operating practices, relate to those for superheater tubes in steam boilers. As a starting point for making a direct comparison of the predictions of the model when applied to typical operating conditions in (a) steam boilers and (b) sCO_2 recuperators, the REFPROP database [10] was used to calculate the required thermodynamic and fluid properties assuming system values of P=200-250 bar and T=500-750°C. This database provides accurate properties for specific heat, density, viscosity, and thermal conductivity, which are key parameters for heat transfer calculations.

The following differences between the properties of sCO_2 and steam can be noted at these operating (T, P) conditions:

1. The specific heat of sCO_2 is approximately 0.3-0.5x that of steam.
2. The density (ρ) of sCO_2 is approximately 1.5-1.7x that of steam.
3. The thermal conductivity (k) of sCO_2 is approximately 0.6x that of steam.
4. The Prandlt number (Pr) in sCO_2 is approximately 0.6-0.8x that of the steam.

Based on assumed flow rates, hydraulic diameters, D, and these thermophysical properties, the local heat transfer coefficient (h) was calculated using correlations for the Nusselt number (Nu), via:

$$h = \frac{Nu_D(Re_D, Pr)\, k}{D}$$

Preliminary results for oxide growth and exfoliation for sCO_2 flow paths

In order to set up numerical simulations, the operating conditions for proposed sCO_2 Brayton cycle systems were surveyed, and pertinent operating parameters are summarized in Table 3. Since the sCO_2 channels for the high-pressure side and the low-pressure side of the recuperator design considered here are bundled together in layers (Figure 4a), there are no pressure differentials and temperature gradients within the wall between two adjacent channels handling flowing sCO_2 at identical pressure and temperature conditions.

A further complicating factor is that a prime alloy candidate for construction of these recuperators is IN740H, which is a relatively new alloy for which data for scale growth and exfoliation in steam and in sCO_2 are lacking. Based on comparison of laboratory steam and sCO_2 oxidation kinetics for IN740H and the morphologies of the resulting scales with those for austenitic steels after service in steam boilers, it was decided that as a worst case scenario for IN740H, its oxide growth kinetics could be considered to be the same as those of the *inner oxide scale* for alloy TP347HFG in steam. Available isothermal kinetics data indicate that the thickness of the oxide scale would be less than 90 μm after 40kh in steam (Figure 6). Since IN740H forms a single layer of external scale in steam and in sCO_2, it was assumed that when scale failure and exfoliation occurs on this alloy, the entire scale thickness would be lost. This is in contrast to the situation modeled for T347H in steam, where only the outer oxide layer (magnetite) is found to exfoliate. As part of the overall project from which this paper is taken, isothermal sCO_2 oxidation/corrosion exposures are being conducted with measurement of oxide thicknesses to develop the needed kinetic data [14].

In modifying the scale exfoliation model for application to the sCO_2 recuperator design considered it was necessary to simulate scale growth and exfoliation from not only the inside (concave) surface of a flow channel (tube), but also the outside (convex) surface. This change is illustrated in Figure 6, which shows the formation of 50 μm thick oxide scales on cross-sections of the internal and external surfaces of 200 μm thick tubes in Figures 6b and 6c, respectively (metal walls drawn to scale). Due to the very thin walls in these heat exchanger architectures, the resistance of the alloy to high-temperature oxidation is likely to be a paramount consideration. The wall thickness defines the available reservoir of the elements (predominantly Cr) required to form a protective scale, so that the ability of the alloy to rapidly form a slow-growing and adherent scale, thus minimizing consumption of the reservoir, becomes an important property.

Table 3. Typical operating conditions for sCO_2 recuperators as compared to those in a steam boiler superheater tube.

Fluid/Size/Property	*High-Pressure Side sCO_2*	*Low-Pressure Side sCO_2*	*Steam Tubes*
ID pressure [MPa]	20-30	3	25
OD pressure [MPa]	20-30	3	1
Differential P across the metal wall	none	none	24
ID temperature [^oC]	50-750	700-	600
OD temperature	50-750	700-	1,200
Differential T across the metal wall	none	none	600
Cycles full-load to low-load	--	--	Weekly or daily
Cycles full-load to room temperature	180 per year	180 per year	Variable but generally only 2-4 planned outages a year

Cases considered for the numerical simulation of exfoliation at isothermal conditions are shown in Table 4. At high load, the temperature along the channel length was set to 700^{o}C. The oxide scale grows stress-free at high temperatures, but stresses build up during temperature excursions to lower temperatures. Thus, in steam boilers the oxides are likely to reach stress levels at which exfoliation becomes possible during shut-down events for austenitic materials (or start-up events for ferritic materials) when the heat exchangers experience the largest temperature swings.

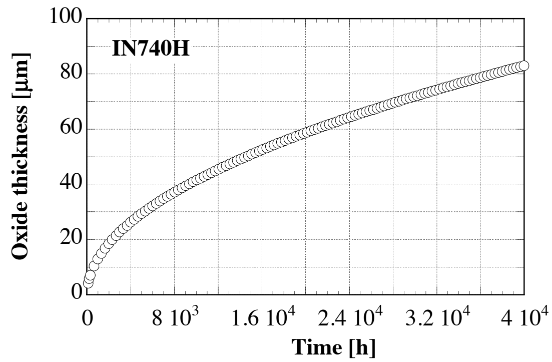


Figure 6. Preliminary data for oxide growth on Inconel 740H at 700°C in the absence of exfoliation (note: growth rate used based on alloy TP347HFG in steam for purposes of developing the model while actual kinetic data is being generated through laboratory exposures).

The coefficients of linear thermal expansion (CTE) of both the metal and oxide scale are very important properties. For chromia, the scale formed on alloy IN740H, the CTE was taken to be 10.75×10^{-6} 1/K [11].

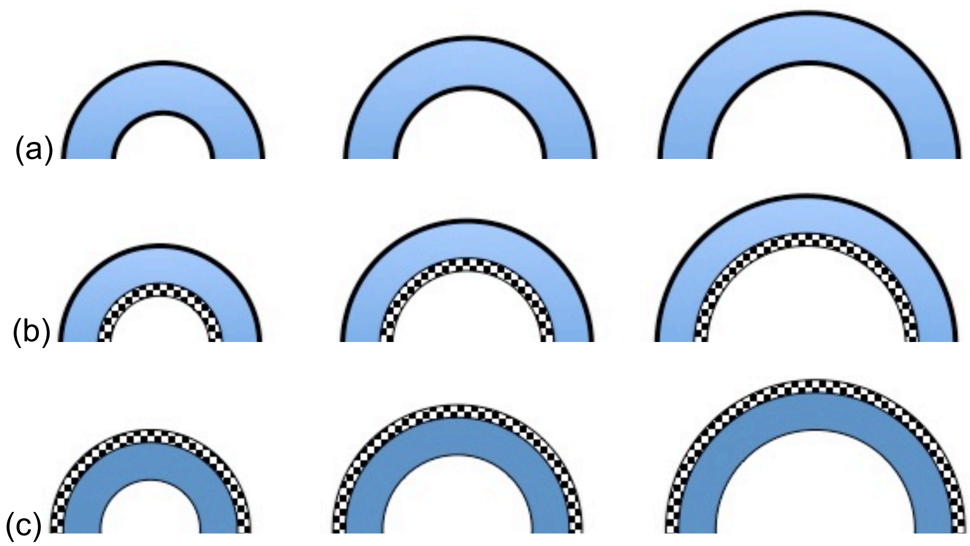


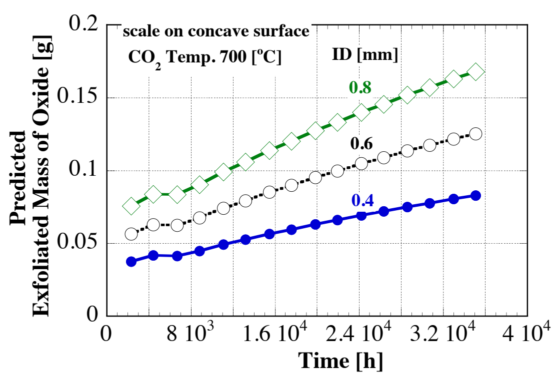
Figure 7. Simplified flow path cross-sections for sCO_2 recuperators: (a) before any oxide scale formation, (b) after formation of a 50 µm oxide scale on the *internal* tube surface, and (c) after formation of a 50 µm oxide scale on the *external* tube surface. Metal dimensions and oxide thickness are drawn to scale.

It should be mentioned that CTE values between 5.7 and 9.6×10^{-6} 1/K were reported in the literature, variation which may be due to differences in the thickness of the films investigated and/or possible anisotropy in the CTE for Cr_2O_3 [11, 12]. The CTE of the alloy was provided by the manufacturer [13]. For these preliminary simulations, shut-down events in sCO_2 recuperators (leading to significant thermal cycles) were considered to occur at 3 month intervals.

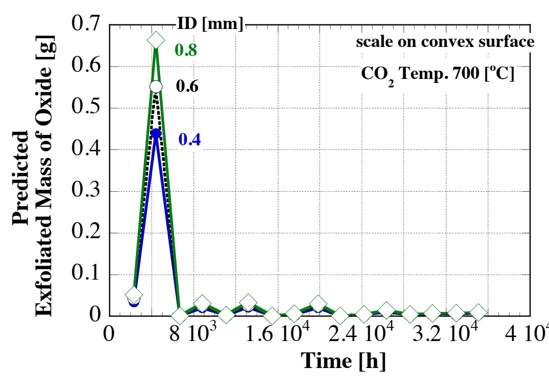
Results for the mass of oxide exfoliated as a function of time over the total length of flow channel considered (500 mm) are presented in Figure 8 for the simulation of alloy IN740H in sCO_2 at 200 bar and 700 ^oC. Compared to amount of scale exfoliated from the much larger surface area of the flow channels (superheater tubes) in steam, Figure 2, the amount of scale lost in one channel of the sCO_2 recuperator configuration considered are miniscule. Nevertheless, the critical factor that determines the potential for blockage is the amount of exfoliant relative to the diameter of the flow channel. Further analysis of the likely fate of scale exfoliated from the internal surfaces as well as from the external surfaces of the channels is necessary before blocking diagrams equivalent to those illustrated in Figure 3 can be drawn for sCO_2 recuperator designs.

Table 4. Cases considered for blockage simulations from exfoliation in sCO_2 at 200 bar and 700 ^oC.

Case	*Metal*	*Location of Oxide Scale*	*ID [mm]*	*OD [mm]*
In1	IN740H	inside	0.4	0.8
In1o	IN740H	outside	0.4	0.8
In2	IN740H	inside	0.6	1
In2o	IN740H	outside	0.6	1
In3	IN740H	inside	0.8	1.2
In3o	IN740H	outside	0.8	1.2



(a)



(b)

Figure 8. Predicted mass of exfoliated oxide scale from a T740H tube after oxidation in sCO_2, using a pure elastic model for the calculating the stresses and strains: (a) scale grown on concave surfaces (inside) and (b) scale grown on convex surfaces (outside). Note: the vertical scales are different.

These results also show that larger amounts of oxides are expected to exfoliate at larger flow path sizes. The model suggests that significantly larger amounts of oxide, approximately 5x, are expected to exfoliate from the convex (outside) surfaces than from the concave (inside) surfaces. This could be simply due to the fact that the surface area available for oxide growth is larger on the outside of the flow channels. In addition, the exfoliated mass exhibits different pattern variations for the oxide grown on convex surfaces from those grown on concave surfaces. For the oxide scale grown on concave surfaces (Fig. 8a) the exfoliated mass of oxide increases steadily, while for the oxide scales grown on convex surfaces (Fig. 8b) the evolution of the exfoliated mass of oxide exhibits nominally a bell-shape curve.

Conclusions and future work

The EPRI-ORNL oxide scale exfoliation model was modified to incorporate the specific properties of sCO_2 and to address particular design features of recuperators being developed for sCO_2 Brayton cycle systems. Preliminary isothermal simulations were made to test the effects of these changes on oxide growth, stress evolution, and scale exfoliation for a generic heat exchanger configuration using alloy IN740H. In the absence of specific oxidation data for IN740H in sCO2 at the time of writing, oxide growth kinetics for the inner layer of scale formed on austenitic steel TP347HFG (which is known to be similar to the single-layered scale formed on IN740H in HP steam) were substituted. The model results for the exfoliated mass of oxide scale in sCO_2 show that significantly larger amounts of oxide, approximately 5x, are expected to exfoliate from the convex (outside) surfaces than from the concave (inside) surfaces. In addition, the exfoliated mass exhibits different pattern variations for the oxide grown on convex surfaces from those grown on concave surfaces. For the oxide scale grown on concave surfaces the exfoliated mass of oxide increases steadily, while for the oxide scales grown on convex surfaces the evolution of the exfoliated mass of oxide exhibits nominally a bell-shape curve, resembling the typical variation observed in steam boiler tubes.

It is important to note that, given the small size of the flow channels and the complex geometries of the heat exchanger designs being considered for sCO_2 recuperators, the thickness of the thermally-grown oxide alone can become a factor of some significance in terms of reducing the flow cross sections or possibly causing blockage, even in the absence of exfoliation. Hence, detailed knowledge of the oxide growth behavior of all the alloys considered for sCO_2 heat exchangers is vital, and is a major goal of the program from which these results were taken.

Results from the model that relate to the potential for blockage following exfoliation were not presented in this paper, since the predictions are very sensitive to specific geometric parameters of the heat exchanger (Sabau et al. 2012), the implications of which have yet to be fully understood. In addition, the creep in the alloy may be important for thin wall recuperator considered (Sabau et al. 2010). While experience with blockage of steam tubes suggests a typical length of channel over which blockage occurs (‘blockage length’) of approximately 5*ID, this may not be the case for the quite different geometries proposed for sCO_2 channels. In addition, the location of blockage sites for oxide grown on -and exfoliated- from the outside surfaces of the sCO_2 channels have yet to be identified. Another important parameter in the blockage model is the frequency of temperature excursions likely to initiate exfoliation. Preliminary data from sCO_2 recuperator OEMs indicate that those heat exchangers would experience more cycling than a superheater in a steam power plant. The factors of importance to the model, such as time between temperature excursions of critical magnitude, need to be clarified before they can be incorporated.

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