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Characterization and Electrochemical Analysis of Acidic Condensate-Induced Corrosion on Aluminized Coating on Steel in Residential Heaters

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Abstract: Condensing furnace residential heaters are starting to replace outdated and less efficient non-condensing units in homes across the US. However, the burning of natural gas in these new units produces acidic gases, which can form acid droplets (H₂SO₄, HNO₃, H₂CO₃, etc.) that are corrosive to low-grade heat exchanger metallic materials. Type-1 aluminized steel has been used in industrial applications, such as marine, heating, and automobile parts, due to its resistance to oxidation at elevated temperature. Many components of the condensing furnaces, including heat exchangers, are made from type-1 aluminized steel. We investigated the interaction of type-1 aluminized steel substrates with two acidic condensate liquids by 500 h corrosion exposures as well as short-term electrochemical impedance measurements. SEM plan and cross-sectional view images revealed damage to the Al-Si rich layer and exposure of the steel substrate. The non-uniform damage in the Al-Si rich layer was likely attributed to a pitting corrosion mechanism and induced by high acidity of the condensate liquid. Electrochemical impedance measurements in an acidic condensate revealed lower corrosion resistance of the aluminized steel surface with a weld line, identifying welded sections as preferential corrosion initiation sites.

Keywords: residential heater; aluminized steel; acidic condensate; corrosion; electrochemical impedance

1. Introduction

The most common space heating equipment in the US residential and commercial buildings are natural gas furnaces, with over 62 million home units in use [1]. These natural gas furnaces can be classified as standard (non-condensing) and condensing furnaces. Most of the units (~75%) are non-condensing standard units, with an annual fuel utilization efficiency (AFUE) of 80% or less [2]. Improving the AFUE of the residential heating units would reduce the fuel consumption and lower the CO_2 emissions. Several approaches to improve AFUE of residential and commercial heating units have been considered. One of the most effective methods for increasing AFUE is to use a condensing furnace setup, which can achieve up to 90% AFUE [3,4].

In condensing furnaces, latent heat is captured from the condensing vapor, which reduces the temperature of the combustion exhaust or flue gas down to about 60 °C. However, one of the disadvantages of reducing the flue gas temperature is the formation of acidic condensates with pH values between 3 and 6. Sulfur, nitrogen and carbon, which are present in natural gas or its combustion processes, can form acidic gases, such as SO₃, SO₂, NO₂, CO₂, etc. [5]. When the flue gas temperature is lower than the dew point of the acidic species, acidic gases can react with water to form corrosive acids, such as H₂SO₄, HNO₃, H₂CO₃, etc. [6]. These acids are corrosive to low-grade steels including carbon steels. To avoid serious corrosion issues, two strategies have been implemented: (1) keeping the



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Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). exhaust temperature above the dew point temperature or (2) using higher grade, corrosionresistant, stainless-steel heat exchangers. The former strategy is not desired as it reduces the overall efficiency of the furnace, and the latter approach has a significant cost penalty due to the use of costly stainless steels. An alternative method is to continuously remove acidic gases during the operation of the heating unit. We recently tested this method by adding a low-cost nano-array monolith trap for acidic gas removal. The design and findings from this setup are described elsewhere [7].

Corrosion of steels arising from condensing gases has been a problem in the industry over several decades [8]. The resistance to atmospheric and elevated temperature corrosion of steels can greatly be improved by applying a uniform layer of aluminum or aluminum alloy coating. The aluminized steel coatings have been used in automobile, heating, marine, and building industries over several decades [9-11]. Aluminized steel is commonly used in automotive exhaust systems as it can withstand the high temperatures and corrosive gases that are present in the exhaust [12,13]. It is also used in heat exchangers, ovens, and other high-temperature applications [14–16]. While the aluminized steel has been used in many different industries and corrosive environments, its use in condensing furnaces for residential heating has only started recently [7]. To investigate the impact of natural gas acidic condensates on the corrosion of type-1 aluminized steel (see Figure 1), the furnace component specimens made of aluminized steel were subjected to the acidic condensate liquids for a period of 500 h at room temperature. In addition, electrochemical impedance measurements were performed to assess short-term corrosion resistance of an aluminized steel surface with and without a weld in an acidic condensate so that preferential sites for corrosion initiation could be identified.



Figure 1. Schematic of an aluminized steel cross-section showing the coating layer and the substrate. The aluminum coating was applied by hot-dipping of low-carbon steel. These coupons were manufactured by AK steel.

2. Experimental Methods

2.1. Corrosion Testing and ICP-OES

Condensate liquids produced by natural gas condensing furnaces during operation are known to be acidic and corrosive to metallic alloys [17–20]. Type-1 aluminized low-carbon steel from a commercial natural gas condensing furnace was obtained for corrosion investigation. The aluminized steel condenser pipes were developed by AK steel. The coating is applied using a continuous hot-dip on both inner and outer surfaces of the pipe to produce a layer with approximately 91% Al and 9% Si composition metallurgically bonded to the low-carbon steel substrate, as shown in Figure 1 above.

Corrosion testing of the condenser pipes was carried out using a 1-inch Gamry glass cell apparatus, see Figure 2 below. The alloy was placed on a flat plastic stage and secured to the Gamry glass apparatus by a stainless-steel clamp. The bottom of the Gamry glass apparatus was sealed with Teflon. This allowed the condensate liquid to be contained inside the apparatus during the corrosion test and in contact with the surface of aluminized steel at room temperature. Immersion tests are commonly used to evaluate the corrosion behavior of metallic materials in previous works [21–25].



Figure 2. Corrosion exposure experimental setup, side view of aluminized steel samples immersed in a condensate solution (**left**), and top view of the setup before immersion in a condensate (**right**).

The condensate liquids from the furnace were collected at two different heating outputs: 65,000 and 80,000 British thermal unit per hour (Btu/h) during a steady-state operation. For more information on the furnace testing setup and condensate liquid collection, refer to References [7]. A total of 100 mL of the condensate liquid was added to the Gamry glass apparatus with the top covered with Parafilm during the exposure tests to minimize evaporation. The alloy surface was exposed to the static condensate liquid for 500 h. After the exposure, the alloy surface was rinsed with DI water. The exposed alloy samples were cross-sectioned, polished, and characterized using a scanning electron microscope (SEM) (Tescan MIRA3) equipped with energy-dispersive X-ray spectroscopy (EDS).

Inductively coupled plasma-optical emission spectroscopy (ICP-OES), from ThermoFisher Scientific 7400 instrument, was performed on a post-exposure condensate solution to measure the amount of iron and aluminum ions in the solution. Furnace condensate samples were analyzed using ICP-OES with a ThermoFisher Scientific 7400 instrument. The samples were mixed with 2% nitric acid (HNO₃) solution in a 1:1 volume ratio. The prepared mixture was then filtered using a 0.45 μ m syringe filter. Calibration standards (0–50 μ g/mL) for aluminum (Al) and iron (Fe) were prepared from 1000 μ g/mL stock solutions, purchased from High Purity Standards (North Charleston, SC, USA). ICP-OES data were collected for optical emission lines 238.2 nm (Fe) and 309.3 nm (Al).

2.2. Electrochemical Impedance Spectroscopy (EIS)

EIS measurements were made for two aluminized steel sheets, one with and another without weld line and conveniently denoted as 'plain' and 'with-weld' samples, using 65,000 Btu/h condensate (also referred to as 65K Btu) with increasing time. An insulating tape with a circular opening area of 0.124 cm² ($\emptyset = 0.4$ cm) was used to set the exposure area on the plain and with-weld samples, as shown in Figure A1 in Appendix A. The exposure area was faced upward in ~40 mL 65K Btu solution contained in a glass cell, where Pt sheet with ~5 cm² area and reference mercury surface electrode (MSE) with saturated KCl were also placed in a three-electrode configuration. The potentiostat utilized in this work is VMP-300 from BioLogic. For each measurement, corrosion potential was recorded for 1 h, and the potential amplitude of ±10 mV with respect to the last corrosion potential was applied over the frequency range of 200 kHz to 5 mHz during EIS. The Btu solutions for EIS were open-to-air at room temperature. For impedance data fitting, Zview software 4.0g (Scribner Associates Inc., Southern Pines, NC, USA) was used.

3. Results and Discussion

Furnace condensate liquid samples were collected from a commercial condensing furnace. The setup followed the instrumentation standards described in ANSI/ASHRAE 103-2017 [26], which were also described elsewhere [7]. Two different furnace heating outputs were used, 65,000 and 80,000 Btu/h. According to a previous report, the 80,000 Btu/h condensate contained low concentrations of formate (14 ppm), sulfate (<2 ppm), and nitrate (<2 ppm), while the 65,000 Btu/h condensate did not contain any detectable amounts of the three species [7]. About 500 mL of condensate liquid was collected from each heating output and was stored in a refrigerator before use in the corrosion exposures. Table 1 shows the pH values measured before and after 500 h corrosion exposures for each heating output case. Acidic pH values, approximately 3.4 and 3.8, were recorded for both 65,000 and 80,000 Btu/h cases before the corrosion exposures [7]. The acidity of the condensate liquids was notably higher after the 500 h corrosion exposures. This indicates a possible reaction between the alloy surface elements and the acid present in the condensate solution, according to the general reaction below:

$$M_{alloy} + HA_{(aq)} \rightarrow MA_{(aq)} + \frac{1}{2}H_{2(g)}; \ M = metal, \ HA = acid$$
 (1)

Table 1. ICP-OES and pH values of condensate solutions before and after corrosion exposures. The four measurements were made for each condensate sample, and the standard deviations are reported in ().

	65K Btu/h Condensate	65K Btu/h Post-Corrosion	80K Btu/h Condensate	80K Btu/h Post-Corrosion
Fe (µg/mL)	0.00 (±0.01)	0.00 (±0.01)	0.00 (±0.01)	0.62 (±0.06)
Al (µg/mL)	0.00 (±0.01)	15.8 (±0.31)	0.00 (±0.01)	$10.7~(\pm 0.71)$
pН	2.94 (±0.1)	5.66 (±0.1)	3.07 (±0.1)	5.77 (±0.1)

Table 1 also shows the Fe and Al concentrations before and after the corrosion exposures. The Fe and Al concentration values were obtained from ICP-OES measurements as described above. The presence of Al in the condensate liquid after the 500 h exposure indicates the corrosion reaction occurring on the surface of the sample. Iron concentration was undetectable in the 65K Btu/h condensate, while only a small amount was detected in the 80K Btu/h condensate after the corrosion exposures. The detection of Fe in the 80K Btu/h post-corrosion condensate indicates a potential breach of the Al-Si rich protective coating. SEM plan and cross-sectional view images and elemental mapping were carried out to assess the integrity of the Al-Si coating after the exposure to the condensate liquids.

Figure 3 presents three cross-sectional locations in type-1 aluminized steel and five spots in the cross-sections selected for EDS composition analysis. The left image exhibits Al-rich coating and Fe-Al alloy layer on the steel substrate, while another location of Al coating and Fe-Al alloy layer is shown in the center image. A random location within the steel substrate in the right image was also selected for EDS spot analysis. The mass percents of the five spots, from EDS data, are summarized in Table 2. Spot A within the bulk coating area contained 2 wt.% Si, while spot B adjacent to the alloy layer showed much higher Si content (~38 wt.%) along with moderate Fe content (~5 wt.%). The mole ratio of Fe and Al in spot C is approximately 1:4. Based on information in a previous report, the alloy layer is presumed to be a mixture of Fe₂Al₅ and Fe₄Al₁₃ [27]. Spots D and E in the steel substrate of the right image revealed ~2 wt.% Co and varied amounts of Ti and Ni.

Figure 4 shows a plan view SEM image and elemental maps of the 80,000 Btu/h case after 500 h exposure. Two spots where the Al-Si coating appeared to be damaged were observed possibly due to a pitting corrosion mechanism, Figure 4a–c. Figure 5 shows a cross-sectional SEM image and associated elemental mappings of the aluminized steel condenser heat exchanger after exposure to the acidic 80,000 Btu/h condensate. The figure shows a region of the exposed area where pitting corrosion occurred within the Al-Si

coating layer. While the steel substrate was not breached during the 500 h exposures, the depth of pitting attack was up to 90% of the Al-Si thickness. It is possible that pitting attacks in other locations might have fully penetrated the Al-Si layer.



Figure 3. Three cross-sectional locations of type-1 aluminized steel and five spots (A–E) where EDS spot analysis was performed.

Table 2. Elemental compositions of the spots in Figure 3. For spot C, the atomic percents of Fe and Al are 15.4 and 61.4, respectively.

	Mass Percent for Each Location					
Elements	Spot A	Spot B	Spot C	Spot D	Spot E	
Fe	0.24	5	27.4	Balance	Balance	
Al	Balance	46.3	52.8	-	-	
Si	2	38	7.7	-	-	
Ti	0.48	0.27	0.01	3.2	0.35	
Ni	0.24	0.51	0.68	0.33	0.43	
Со	-	0.19	0.49	2.2	2.11	
Minor elements		Nb and Zr		Nb	-	

Figure 6 presents a cross-sectional SEM image and associated elemental mappings of aluminized steel after 500 h immersion in 65,000 Btu/h. The images show a region where the Al-Si layer was damaged by pitting corrosion. The absence of Al and Si along the pitting attack is shown in Figure 6c,d. The pitting corrosion damage to Al-Si layer was deep enough to expose the steel substrate as seen in Figure 6e. On the SEM/EDS plan view observation presented in Figure A2 in Appendix A, oxygen was present on both Al-rich coated and Fe-rich welded surfaces, implying that corrosion occurred not only on Al-Si layer, but also on welded regions.

Impedance spectra of plain and with-weld surfaces in 65K Btu solutions are plotted for two immersion times, 11 and 19.5 h, in Figure 7a–d. The plot shapes of plain surface are characterized by two impedance arcs, while three arcs, including one at high-frequency regions (Figure 7b,d), were observed in the plots of with-weld surface. In general, greater impedance scales are indicative of higher corrosion resistance or lower corrosion susceptibility in EIS measurements of metallic alloys in aggressive solutions [28,29]. Therefore, greater impedance scales of plain surface are likely associated with lower corrosion rate of the surface than with-weld surface. The corrosion potentials where EIS measurements were made are also plotted as a function of immersion time in Figure 7e. The corrosion potentials were distinctively more noble in with-weld surfaces, which suggests that the surface materials, including Al, steel, and/or metallic oxides/hydroxide, in contact with 65K Btu solution were quite different between the plain and with-weld surfaces. Corrosion potentials in 0.5 wt.% NaCl + 3.5 wt.% (NH₄)₂SO₄ solutions, a simulated acidic rain, were higher in uncoated steel than in Al-coated surface [30], which supports the idea that exposed steel surface would cause more noble corrosion potential. Therefore, the exposed steel along the weld (as shown in Figure A2 in Appendix A) was considered as the primary factor that drove more noble corrosion potential of with-weld surface.



Figure 4. (a) Field view SEM electron image after 500 h room temperature exposure in 80,000 Btu/h condensate with (**b**–**f**) elemental mapping associated with image (**a**).



Figure 5. (**a**) Polished cross-sectional view SEM electron image after 500 h room temperature exposure in 80,000 Btu/h condensate with (**b**–**f**) elemental mapping associated with image (**a**).



Figure 6. (**a**) Polished cross-sectional view SEM electron image after 500 h room temperature exposure in 65,000 Btu/h condensate with (**b**–**f**) elemental mapping associated with image (**a**).

Based on the initial analysis on impedance spectra and corrosion potential transients, two impedance circuits for the plain and with-weld surfaces are proposed in Figure 8a,b, with the schematics of sample cross-sections explaining the respective positions of resistive and capacitive components. In the plain surface sample, Al- and O-rich defective film is presumed to be partially soaked with 65K Btu solution and to behave as a parallel pair of resistor and capacitor ($R_{\rm film}//CPE_{\rm film}$), and the interface of soaked top film and underlying Al-rich coating is modeled using $R_{\rm intf}//CPE_{\rm intf}$. In this model (Figure 8a), surface corrosion resistance is evaluated by the sum of $R_{\rm film}$ and $R_{\rm intf}$ as the corrosion of metallic components, Al and Fe, is presumed to occur at the interfaces and controlled by the transfer of charged species through the oxide films. In the meantime, the with-weld surface is assumed to have a highly porous and fully soaked corrosion product film above the defective film and coating layers, adding $R_{\rm crp}$ and $CPE_{\rm coat}$ to the two parallel R//CPE pairs for impedance data fitting. In this case, the sum of $R_{\rm crp} + R'_{\rm film} + R_{\rm intf}$ was used for corrosion resistance assessment. $R_{\rm crp}$ is considered as an additional layer influencing the transfer of charged species during corrosion.

Individual and sum of fitted resistances are plotted as a function of immersion time for the plain and with-weld surfaces in Figure 8c,d, respectively. R_{sol} is solution resistance, R_{film} and R'_{film} are defective film resistances, R_{intf} is interface resistance underneath the defective films, and R_{crp} is corrosion product resistance. CPE_{film} and CPE_{intf} account for non-ideal capacitance behaviors associated with film and interface resistance elements. CPE_{coat} is the non-ideal capacitive behavior of the [Al-rich, Si] coating layer adjacent to the weld. Individual and sum of resistance values from impedance data fitting for (c) plain and (d) with-weld surfaces with increasing immersion time. Other fitted electrochemical parameters and fitting quality data are summarized in Tables A1 and A2 in Appendix A.

The resistances of defective films (R_{film} and R'_{film}) were lower in the with-weld surface presumably due to the higher solution saturation associated with more defects created in the layer by the welding process. R_{crp} values were low as expected from a highly porous layer. In Figure 8e, the sum of resistances, representing the surface corrosion resistance, was lower in the with-weld surface and became only 28% of the plain surface at 19 h immersion. These results confirm that corrosion attack would preferentially occur near/at the welded surface. The interfacial resistances were slightly higher in [Al-rich, Si] than in Al-Si-Fe fusion layer. In the case of Al without Si, corrosion resistance in 3.5 wt.% NaCl was higher in Al-Fe than in Al [31], which is opposite to the current result. A future corrosion analysis comparing Al-Si and Al-Si-Fe would be needed to understand an effect of Fe in the condition more relevant to this work.



Figure 7. Electrochemical data of plain and with-weld aluminized steel surfaces in 65K Btu solution. Impedance plots after (**a**,**b**) 11 h and (**c**,**d**) 19.5 h immersion. (**b**,**d**) Impedance data in smaller x and y scales. Note that with-weld data in (**a**) were plotted to 0.012 Hz due to low-frequency noise. The low frequency of the other impedance data was 0.005 Hz. (**e**) The corrosion potential values associated with impedance measurements.



Figure 8. Equivalent circuit models illustrated in schematic cross-sections of (**a**) plain and (**b**) withweld surfaces. R_{sol} : solution resistance, R_{film} and R'_{film} : defective film resistances, R_{intf} : interface resistance underneath the defective films, and R_{crp} : corrosion product resistance. CPE_{film} , CPE_{intf} , and CPE_{coat} : constant phase elements associated with the defective film, interface, and [Al-rich, Si] coating layer. All resistance data in (**c**,**d**) were from the fitting results with Chi-square lower than 0.00089.

The estimated capacitance values of plain and with-weld surfaces are plotted as a function of immersion time in Figure 9. The interface capacitances were not significantly different between the plain and with-weld surfaces, while the film capacitances were greater in the with-weld case. The discrepancy of film capacitance could be attributed to film permittivity and thickness that must have been different between the two samples. For the plain and with-weld samples, the interface capacitances were greater than the film capacitances (>1 order). Similar to these results, smaller film capacitances were previously reported in type-2 aluminized steel corroding in a neutral electrolyte simulating natural water [32]. The coating capacitance associated with the layer away from the weld was the smallest among the estimated capacitances. This is probably because the major portion of charged species transfer during corrosion occurred within the welded region; therefore,

only a small portion of charged species would participate in the capacitive behavior of the non-welded coating layer.



Figure 9. Estimated capacitance of *CPE* elements with increasing solution immersion time for the plain and with-weld surfaces. The numerical capacitance values are summarized in Tables A1 and A2 in Appendix A.

4. Summary

Two acidic condensates, which were collected from a residential heating unit, were used to test their corrosivity towards type-1 aluminized steel samples. The pH values of the two condensates were 2.94 and 3.07 for the 65,000 and 80,000 Btu/h, respectively. The samples immersed at room temperature for 500 h showed significant damage to the aluminized steel coating as indicated by the detection of aluminum in the post-corrosion condensates as well as SEM images. The cross-sectional SEM images showed pitting corrosion that penetrated Al-Si coating and exposed the steel substrate. For the 65,000 Btu/h case, the Al-Si rich coating layer was breached as seen in the cross-sectional SEM and elemental mapping images. Electrochemical impedance spectroscopy (EIS) was used to compare the corrosion resistance of plain and with-weld aluminized steel surfaces immersed in 65K Btu solution. According to the analysis of EIS data, overall corrosion resistance is expected to be lower in with-weld surfaces, where the welded region was presumably the major location associated with the corrosion process. Finally, we showed that EIS can be used as a predictive method to understand the corrosion susceptibility of type-1 aluminized steel in acidic liquids when long-term exposure tests are not immediately available.

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Appendix A



Figure A1. Photo images of tape-masked surfaces for (a) plain and (b) with-weld surface specimens to support the experimental setup description. The surface area is 0.124 cm^2 .



Figure A2. (a) Field view SEM electron image after 500 h room temperature exposure in 65,000 Btu/h condensate with (**b**–**f**) elemental mapping associated with image (a). In this image, the smoother surface (right side of the image) shows a region where the steel substrate was exposed along the weld line.

Table A1. Fitted resistance and CPE values from impedance data of plain surface. For all data, n of CPE_{intf} is fixed to 1 to avoid the value greater than 1 from automated fitting [33].

Immersion Time		2 h	5.5 h	11 h	19.5 h
$E_{\rm corr}/{\rm mV}_{\rm MSE}$		-833	-823	-806	-782
R _{sol} /ohm		566	403	270	216
$R_{\rm film}/\rm ohm\cdot cm^2$		6252	5667	5890	6902
$R_{\rm intf}/\rm ohm\cdot cm^2$		1518	1483	1546	1761
CPE _{film}	Capacitance/F·cm ^{−2}	$1.13 imes 10^{-5}$	$1.6 imes10^{-5}$	$2 imes 10^{-5}$	$2.38 imes 10^{-5}$
	n	0.95	0.95	0.94	0.93
CPE _{intf}	Capacitance/F·cm ^{−2}	$2.5 imes 10^{-3}$	$3.15 imes 10^{-3}$	$3.55 imes 10^{-3}$	$3.88 imes 10^{-3}$
	n	1	1	1	1
Chi-square		$8 imes 10^{-4}$	$6.9 imes10^{-4}$	$5.3 imes 10^{-4}$	$3.5 imes10^{-4}$

Immersion Time		5.5 h	11 h	19.5 h
$E_{\rm corr}/{\rm mV}_{\rm MSE}$		-646	-638	-643
R _{sol} /ohm		849	503	731
$R_{\rm crp}/{\rm ohm}\cdot{\rm cm}^2$		146	72	113
$R_{\rm film}/\rm ohm\cdot cm^2$		1027	1227	996
$R_{\rm intf}/\rm ohm\cdot cm^2$		2550	2351	1301
CPE _{coat}	Capacitance/F·cm ^{−2}	$1.38 imes10^{-8}$	$4.02 imes 10^{-8}$	$2.19 imes 10^{-8}$
	n	0.95	0.92	0.94
CPE _{film}	Capacitance/F·cm ^{−2}	$1.02 imes 10^{-4}$	$1.16 imes10^{-4}$	$1.31 imes 10^{-4}$
	n	0.74	0.79	0.78
CPE _{intf} -	Capacitance/F⋅cm ⁻²	$4.52 imes 10^{-3}$	$5.32 imes 10^{-3}$	6.62×10^{-3}
	n	0.91	0.85	1
Chi-square		$2.9 imes10^{-4}$	$8.9 imes10^{-4}$	$2 imes 10^{-4}$

Table A2. Fitted resistance and CPE values from impedance data of with-weld surface. For the data with 19.5 h immersion time, CPE_{intf} is fixed to 1 to avoid the value greater than 1 from automated fitting [33].

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