

Tensile Properties of Haynes Alloy 230 and Inconel 617 after Long Exposures to LiF-22CaF₂ and Vacuum at 1093 K

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As a part of a study of a space-based thermal energy storage system utilizing the latent heat of fusion of the eutectic salt LiF-20CaF₂ (mole %), the two wrought Ni-base superalloys Haynes alloy 230 and Inconel 617 were subjected to molten salt, its vapor, and vacuum for periods as long as 10,000 h at 1093 K. Following exposure, the microstructures were characterized, and samples from each superalloy were tensile tested between 77 and 1200 K. Neither the structure nor mechanical properties revealed evidence for additional degradation due to exposures to the salt. Although some loss in tensile properties was noted, particularly at 77 K, this reduction could be ascribed to the influence of simple aging at 1093 K.

Keywords

Inconel 617, Haynes alloy 230, heat treatment, long term exposure, superalloy, tensile properties, thermal energy storage

1. Introduction

A STEADY, reliable, and efficient source of heat and electrical energy is a prime requirement for any manned, earth-orbiting space station. A solar dynamic system that can gather sunlight, absorb and store this energy, and power an electrical generator is capable of fulfilling this task and was considered for this application (Ref 1). Problems associated with energy storage could be particularly severe as the heat of fusion from the liquid to solid phase transformation of LiF-20CaF₂ (mol%) eutectic, which melts at 1043 K, is viewed to be an ideal means to store energy. Because molten fluorides tend to be very corrosive, long-term encapsulation of the salt might be difficult.

The initial design (Ref 2) of the space-based solar dynamic system was established with a relatively low temperature Brayton Cycle heat engine operating at approximately 1015 K, in part, to allow usage of the conventional Co-base superalloy Haynes* alloy 188 (alloy 188) for salt containment. This choice was based on results from previous compatibility experiments with LiF-superalloy combinations (Ref 3-6) and on the excellent long-term 1000 to 1200 K creep rupture strength of alloy 188 in combination with ease of fabricability and weldability.

Concurrent with the choice of energy storage salt and containment alloy, compatibility studies were started at Boeing (Ref 7), AiResearch (Ref 8, 9), and the Lewis Research Center (Ref 10-14) to provide reassurance that alloy 188 would be able to withstand the corrosive attack of the LiF-CaF₂ eutectic. The conclusion from these efforts is that alloy 188 can successfully contain the molten fluoride salt in a space environment. However in the beginning of the program, concern was raised about the loss of room-temperature ductility after long-term exposure

*Haynes is a registered trade name of Haynes International, Kokomo, IN, 46904-9013.

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between 800 and 1300 K (Ref 15). Therefore, at the suggestion of Haynes International, Haynes alloy 230 (alloy 230) was included as an alternative high-temperature material during the initial portion of these studies. Also at the start of this research, another study (Ref 7) indicated that the International Nickel Company's Ni-base Inconel** 617 (IN 617) might likewise be suitable; hence compatibility experiments involving this alloy were also initiated.

Although the effects of 400 to 22,500 h, 1093 K salt and/or vacuum exposures on alloy 188 were documented (Ref 10, 11, 14) and the influence of 4900, 10,000, and 22,500 h heat treatments in air at 1093 K on alloy 188, alloy 230, and IN 617 were published (Ref 12, 13), the efforts on the properties of alloy 230 and IN 617 after relatively long-term 1093 K exposures to the LiF-CaF₂ eutectic were not made public. Specifically, this paper summarizes the behavior of IN 617 after exposures of 400, 2500, and 7914 h, while results after 10,000 h of exposure are presented for alloy 230. This represents the entire collection of data for salt-exposed IN 617 and serves as an extension for alloy 230 exposed for 400 and 2500 h (Ref 10). The following discusses the structure and tensile properties between 77 and 1200 K of alloy 230 and IN 617 after salt exposures. Data from vacuum-exposed materials are also given to serve as a baseline for comparisons.

2. Experimental Procedures

Approximately 1.27-mm-thick by 0.6-m by 1.2-m sheets of alloy 230 and IN 617 were purchased for this study. From this stock, bread-pan shaped capsules about 280-mm-long by 140-mm-high by 115-mm-wide with matching lids were fabricated by a combination of bending and welding without filler. Racks to hold about 50 tensile test specimens during exposure were constructed by cutting slots into two parallel inverted "L" shaped pieces held apart by stays (Ref 10). Pin and clevis tensile-type test samples, 82.5-mm-long by 12.4-mm-wide with a 25.4-mm by 6.3-mm gage section, were punched from the alloy sheet with the gage length parallel to the sheet rolling direction

**Inconel is a registered tradename of INCO Alloys International, Inc., Huntington, WV, 25720

for alloy 230. Due to a misinterpretation of the sheet orientation, the IN 617 samples were punched with their gage lengths perpendicular to the sheet rolling direction. In all cases, the same heat of alloy was used to make both the bread pans and tensile specimens.

The chemistry and vendor for each material can be found in Table 1. The Ni-base alloy 230 was supplied in a solution-treated condition, whereas the Ni-base IN 617 was purchased in the cold-rolled and annealed state. IN 617 sheet possessed a normal silver metallic sheen, but alloy 230 had a dull, oxide-like gray color due to the black anneal and pickling surface finish processing. Examination of this alloy by both metallography and x-ray diffraction techniques failed to reveal the presence of oxides on the surface. Polished and etched metallurgical sections revealed that alloy 230 (Fig. 1a, Ref 10) had reasonably uniform, equiaxed grain structure about 35 μm in diameter and a relatively uniform, but sparse, dispersion of second-phase particles. This material did not have a smooth sheet surface; rather alloy 230 contained pits as deep as 10 μm from the pickling process (Fig. 1a, Ref 10). IN 617 had a relatively flaw-free, smooth surface; however, examination of the microstructure revealed a thin layer of approximately 10 μm grains at the sheet surfaces and a much larger grain diameter ($\sim 85 \mu\text{m}$)

Table 1 Characterization of starting materials

Material	Vendor	Heat or lot	Composition, wt%
Alloy 230	Cabot Corp.	1830557171	0.004B-0.3Al-0.10C-22.00Cr-1.22Fe-0.009La-0.61Mn-1.29Mo-0.01P-<0.002S-0.39Si-14.01W-Ni
IN 617	INCO Alloys International, Inc.	XX0130UK	1.05Al-0.002B-0.06C-13.46Co-21.83Cr-0.08Cu-1.66Fe-0.14Mn-9.25Mo-<0.001S-0.11Si-0.24Ti-Ni

within the sheet interior (Fig. 1b, Ref 12). Unlike alloy 230, as-received IN 617 had little evidence of any second phases.

After being cleaned by hot vapor degreasing in trichloroethane followed by immersion within an ultrasonic bath, the tensile specimens were individually weighed and measured. Samples were then placed into specimen racks and recleaned along with bread pans and lids. With a rack of specimens on the bottom of the capsule, about 2.6 kg of nominally LiF-22CaF₂ chunks (Table 1) were poured into the bread pan. After the second rack was placed on tabs within the capsule, the lid, which contained two approximately 3 mm holes, was gas-tungsten-arc welded in an argon atmosphere to the main body. This assembly was then slowly heated to 1093 K under vacuum (8 h from room temperature to 993 K, 2 h to 1093 K, 1 h hold at temperature, and a furnace cool) to remove dissolved gases and water from the salt. Directly after this heat treatment, both holes in the lids were closed by electron-beam welding.

Bread-pan capsules with a third rack of specimens on top were heat treated at 1093 K in a cryogenically pumped vacuum of approximately 1.3×10^{-4} Pa or better. None of these exposures were continuous; all experienced shut downs due to loss of electrical power or cooling water, vacuum leaks, regeneration of the cryopumps, etc. While little difficulty was encountered with gas-tungsten-arc welds in the alloy 230 bread pan, several salt traces were found near the welds on an IN 617 bread pan after completion of its scheduled 400 h vacuum heat treatment at 1093 K. Once the inability to produce acceptable welds in IN 617 became apparent, the remaining IN 617 bread pans were inspected after giving them a 24 h, 1093 K vacuum anneal prior to starting the long-term exposures. In spite of visual inspection efforts, salt leaks around welds in IN 617 were always found after this heat treatment. Because of this contamination, these areas simply could not be rewelded close. Instead larger shells (approximately 300-mm-long by 160-mm-high by 130-mm-wide with matching lids) were fabricated from Ni-201. The IN 617 capsules were then placed inside the Ni shells, and several small holes were drilled through the IN 617 lid. The

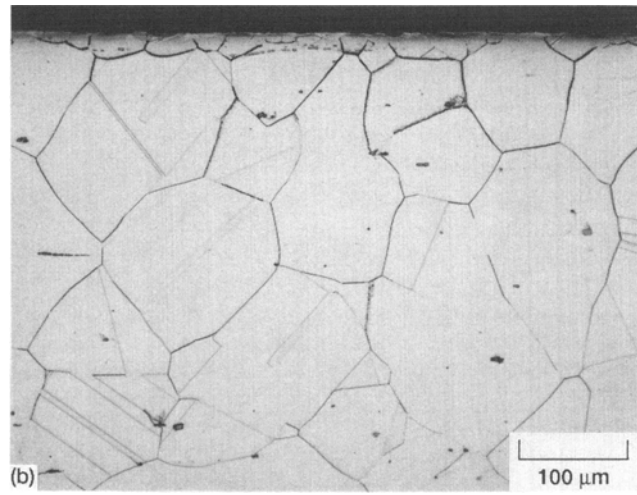
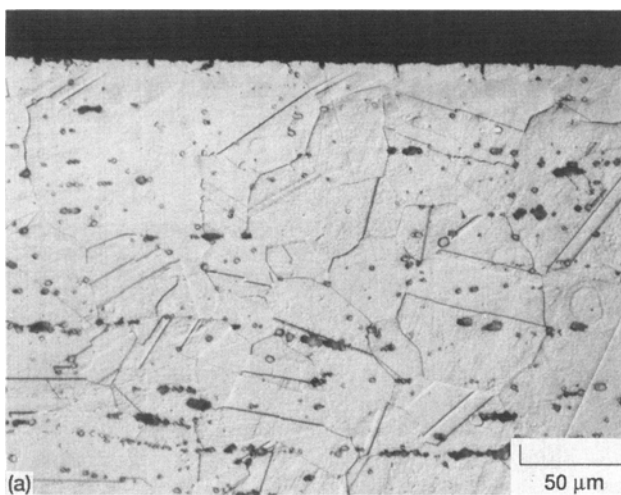


Fig. 1 Photomicrographs of the near surface microstructure of the as-received alloys. (a) Haynes alloy 230 and (b) Inconel 617. Haynes alloy 230 was immersion etched in a mixture of 97 parts HCl and 3 parts H₂O₂; Inconel 617 was electrolytically etched at 4 V, 0.5 amp for two to three seconds in a mixture consisting of 95 ml H₂O and 5 ml HCl.

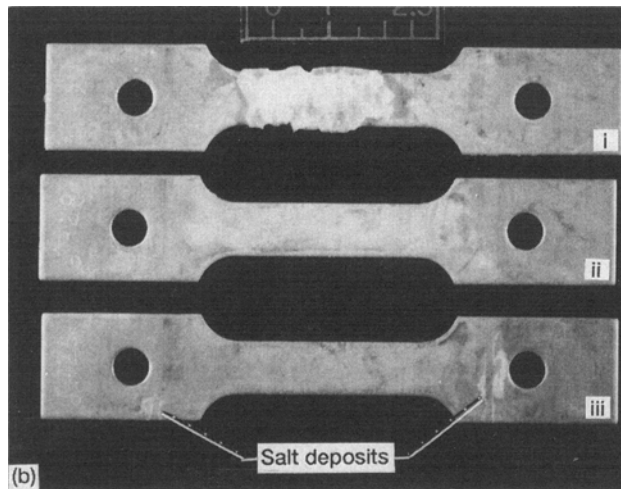
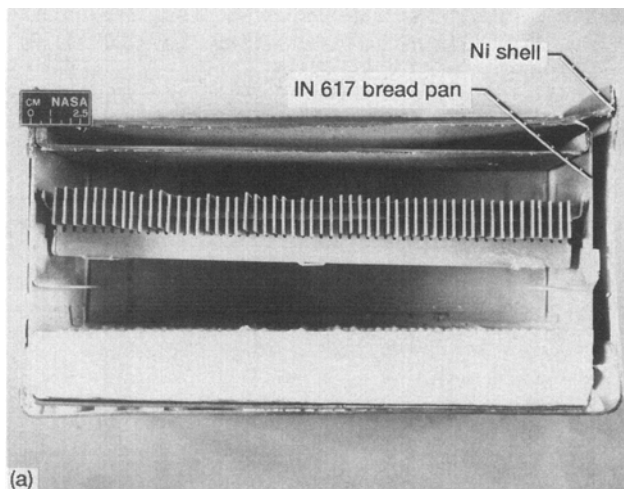


Fig. 2 Photographs of (a) the opened Ni shell and Inconel 617 bread pan capsule after being heat treated in vacuum at 1093 K for 7913.8 h and (b) molten-salt exposed specimens. Part (i) is as-exposed condition, (ii) is after scraping to remove bonded salt, and (iii) is after a 1 h, 1093 K vacuum heat treatment.

Ni-201 lid was then welded to the Ni shell, and the assembly was given the same stepped vacuum heat treatment noted above to remove dissolved gases and water. Upon completion of this heat treatment, the vent holes in the Ni lid were electron-beam welded closed. After the racks of IN 617 tensile specimens slated to be vacuum exposed were placed on top of the Ni shell, the assembly was placed in the vacuum furnace and heat treated without further incident.

Upon completion of the planned corrosion experiment, the capsules were cut open and photographed, and several samples of frozen salt were taken for chemical analysis. The LiF-22CaF₂ encapsulated specimens were freed by breaking the salt with a hammer, gently tapping the shoulder sections, and manually manipulating individual samples. Prior to measuring and weighing, these specimens were wiped with a clean, lint-free rag whereas visible spots of salt on the vapor-exposed samples were removed by scratching with a gloved hand. The vacuum-annealed samples were not cleaned prior to postexposure measurements.

Triplicate tensile tests were conducted under contract at the Cortest Laboratories, Inc. (NAS3-25759) on LiF-CaF₂ eutectic salt-exposed, vacuum-annealed, and as-received specimens. Testing was undertaken in universal screw-driven machines at a nominal strain rate of 8.3×10^{-5} /s through the 0.2 percent yield; beyond this strain, the deformation rate was increased to 2.1×10^{-3} mm/s and held until failure. Strain was measured via a mechanical extensometer directly attached to the gage section. The testing atmosphere varied with temperature. At 77 K, specimens were immersed in liquid N₂. At 298 K, tests were conducted in air. At and above 750 K, measurements were carried out in a 0.006 Pa or better vacuum. Typical test data included 0.02 and 0.2 percent offset yield strengths, ultimate tensile strength (UTS), and elongation at failure; all mechanical properties were calculated on the basis of the original (preexposure) dimensions.

Standard metallographic procedures and chemical analyses were utilized to characterize the starting, exposed, and tensile-tested materials. Polished metallographic sections of exposed

alloy 230 were electrolytically etched at 4 V and 0.5 to 0.75 amp in solution consisting of 33 mL HNO₃, 33 mL acetic acid, 33 mL H₂O, and 1 mL HF; as-received alloy 230 was immersion etched in a mixture of 97 parts HCl and 3 parts H₂O₂. IN 617 was also electrolytically etched at 4 V, 0.5 amp for two to three seconds in a mixture consisting of 95 mL H₂O plus 5 mL HCl.

3. Results

3.1 Salt/Vacuum Exposures

3.1.1 IN 617.

Upon discovery of leaks in the welds, subsequent LiF-22CaF₂ filled IN 617 bread pans were vacuum sealed in Ni shells and successfully vacuum heat treated for 2500 and 7913.8 h at 1093 K. Figure 2(a) illustrates the geometry of the inside of Ni shell and IN 617 bread pan after side walls were cut away. Salt in the gaps between the Ni shell and IN 617 bread pan indicated that LiF-22CaF₂ leaked out of the bread pan during heat treatment. Such losses, however, did not affect exposure of the lower racks of tensile samples to molten salt as the gage sections were immersed in frozen salt. Furthermore, the upper racks were maintained above the molten LiF-22CaF₂ in the salt vapor. The visible inner surfaces of the bread pans (Fig. 2a) and the vapor-exposed IN 617 specimens were dull-gray matte in color without any visible sign of corrosion. A few vapor-exposed specimens, however, had small droplets of frozen salt on the surface. The racks of vacuum-exposed tensile specimens were basically unblemished and possessed a bright matte silvery metallic finish.

The frozen blocks of LiF-22CaF₂, such as that shown in Fig. 2(a), did not adhere to the capsule walls and were gray-white in color except for the bottom, which was covered with a dark-gray scum. Samples of the salt for chemical analysis were taken from the side and bottom of the block and from regions be-

Table 2 Statistical summary of weight changes found in Inconel 617 tensile-type specimens after various exposures to molten LiF-22CaF₂, salt vapor, or vacuum at 1093 K

Statistical parameters	Weight change (exposed – as-received), mg		
	Molten salt	Salt vapor	Vacuum
400 h			
Average	-5.6	7.8	-1.5
Standard deviation	3.1	1.1	0.4
Maximum	0.8	10.3	-0.7
Minimum	-11.3	5.0	-2.6
2500 h			
Average	-1.9	7.6	-5.5
Standard deviation	2.6	1.4	0.3
Maximum	9.0	10.4	-4.8
Minimum	-6.5	2.9	-6.3
7913.8 h			
Average	0.9(0.0)	-0.6	-10.8
Standard deviation	2.2(2.2)	1.2	0.6
Maximum	6.0(4.5)	2.5	-9.8
Minimum	-3.6(-5.0)	-3.3	-13.1

Note: The data represent results from at least 50 tensile-type specimens where each sample weighed ~15 g and had ~33 cm² of surface area. Statistical parameters in parentheses are values measured after the specimens were given a 1 h, 1093 K heat treatment in vacuum in an attempt to remove some of the tenaciously bonded salt.

tween the tensile specimens, and they did not reveal any evidence of contamination. While many of the IN 617 tensile specimens could be cleanly separated from the frozen salt, pieces of LiF-22CaF₂ were tenaciously bonded to the remaining samples (Fig. 2b, part i). The propensity for adherence of the salt to the specimens increased with time of exposure; about half the IN 617 tensile samples immersed in molten salt for 7913.8 h had bonded LiF-22CaF₂. Severe scraping had to be employed to remove these deposits (Fig. 2b, part ii). In terms of color, the molten-salt IN 617 specimens were dull, mottled-gray in appearance with spotty yellow-brown scum.

The weight change results after various periods of exposure at 1093 K are summarized in Table 2. Analysis of the data for the molten-salt exposed samples is befuddled because small pieces of LiF-22CaF₂ bonded to the IN 617 some of which remained even after a 1 h vacuum anneal at 1093 K (Fig. 2b, part iii). With this in mind, the 400 h result of an average weight loss strongly suggests that minor corrosion of IN 617 is occurring, but evidence of this corrosion becomes hidden in the longer exposures as the salt bonds to the alloy. For the two shorter heat treatments, the vapor-exposed samples slightly gained in weight, whereas the 7914 h anneal produced a very small average weight loss. Vacuum exposures lead to weight losses through volatilization of chromium, manganese, etc., which increased with time of exposure, as noted in Ref 12. Compared to the original surface area (~33 cm²) for each specimen, the relative change after any exposure is negligible (<1 mg/cm²) even in the worst case (Table 2).

Metallurgical specimens to evaluate possible microstructural damage in IN 617 were cut from the bread pans. The material contacted by the molten LiF-22CaF₂ exhibited occasional pits in the unetched state (Fig. 3a), which tended to increase in number and enlarge when etched (Fig. 3b). In general, the salt-

Table 3 Statistical summary of weight changes found in Haynes Alloy 230 tensile-type specimens after 10,000 h exposure to molten LiF-22CaF₂, salt vapor, or vacuum at 1093 K

Statistical parameters	Weight change (exposed – as-received), mg		
	Molten salt	Salt vapor	Vacuum
Average	0.3	21.9 (19.2)	-11.0
Standard deviation	2.1	8.4 (4.8)	1.1
Maximum	6.4	53.6 (25.7)	-9.0
Minimum	-4.9	6.6 (6.6)	-14.7

Note: The data represent results from at least 50 tensile-type specimens where each sample weighed ~15 g and had ~33 cm² of surface area. Statistical parameters in parentheses are values measured after the specimens with obvious salt deposits were removed.

vapor exposed IN 617 had the same appearance as the molten-salt exposed alloy, and no enhanced corrosion was seen at the liquid and/or vapor interface (Fig. 3c). The only unusual behavior was the apparent observation of significant attack of IN 617 at or near welds (Fig. 3d). Such corrosion was seen in weld regions exposed to either the molten salt or its vapor, and the damage occurred in the heat-affected zone as well as in the weld itself. Examination of the vacuum-exposed alloy revealed a slight surface roughening and an occasional pit (Ref 12). After the sheet was etched, M₂₃C₆ carbides (Ref 12) both within the grains and on the boundaries were clearly visible (Fig. 3b), and measurements of the postexposure grain sizes away from the sheet surface gave a value of about 90 μm (Ref 12), which is essentially the same as the as-received diameter.

3.1.2 Alloy 230.

The LiF-22CaF₂ filled alloy 230 bread pan was successfully vacuum heat treated for 10,000 h at 1093 K. Both the vacuum-exposed bread pan and the vacuum-exposed samples had a dull gray color. After the capsule was cut open, the lower rack was found to be completely immersed in frozen salt, which was white in color. The inner surfaces of the bread pan were clean, without any sign of corrosion on either the vapor or liquid exposed areas. The salt-vapor exposed alloy 230 had a matte silver appearance; however, some samples had small droplets of frozen salt bonded to the surfaces. The alloy 230 specimens encapsulated in the frozen salt were broken free by a hammer and mechanical manipulation; they were mottled dull gray in appearance. Chemical analysis of the frozen salt in contact with alloy 230 for 10,000 h at 1093 K did not reveal any contamination.

The weight change data for alloy 230 heat treated in molten LiF-22CaF₂, its vapor, and vacuum for 10,000 h at 1093 K is summarized in Table 3. The results for the molten-salt exposed samples continued the trend set by the 400 and 2500 h of exposures (Ref 10), where little attack seemed to have taken place. The LiF-22CaF₂ vapor-exposed samples had gained a slight amount of weight (<1 mg/cm²), which is probably due to salt accumulations on the surface. The 400 and 2500 h vapor-exposed samples also demonstrated slight increases in weight (Ref 10). Volatilization of chromium, manganese, etc. contributed to weight losses during the vacuum exposure in the same manner as that found after 2500 h heat treatments (Ref 10, 12). As the case for alloy 188 (Ref 10, 11, 14) and IN 617, even in

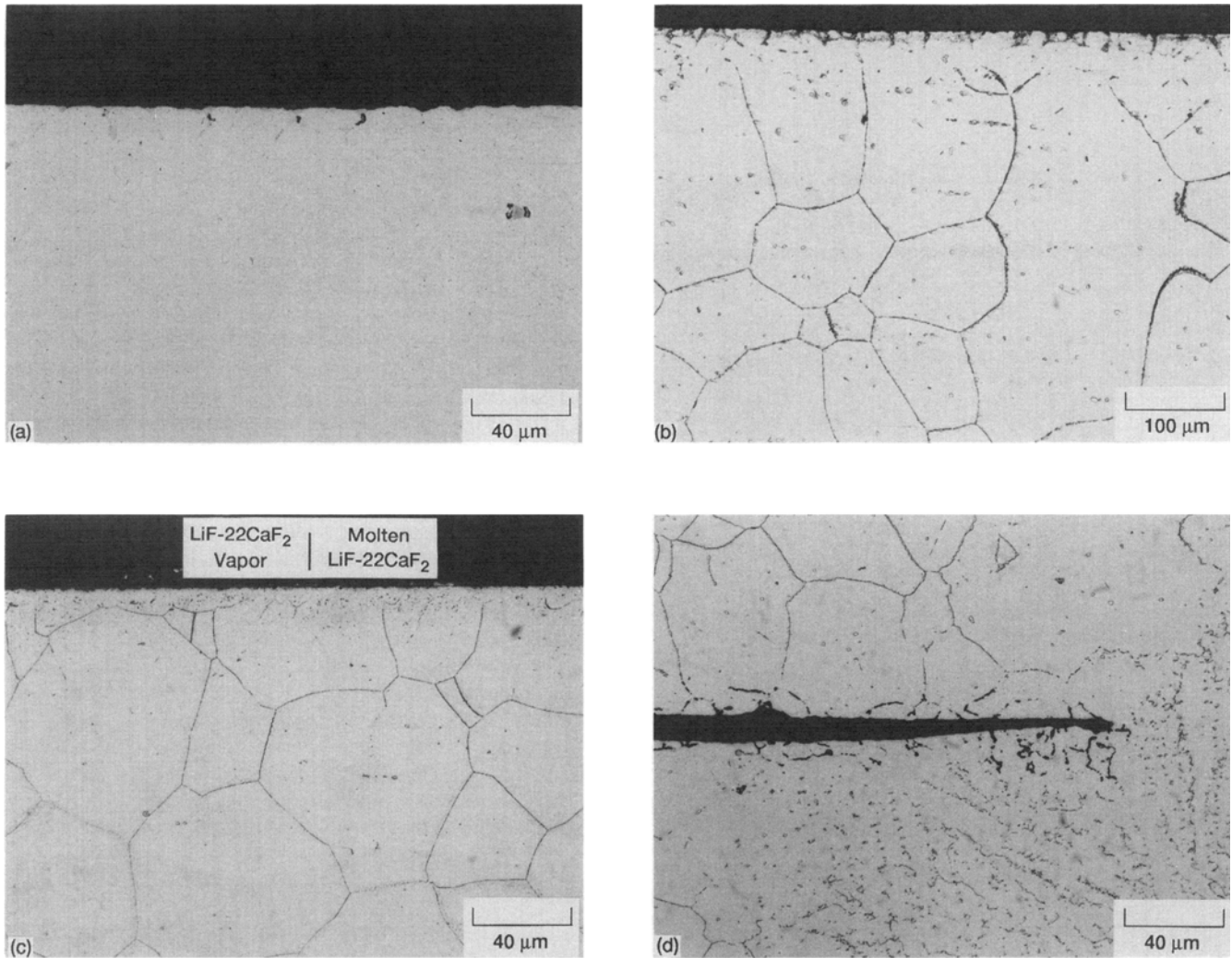


Fig. 3 Photomicrographs of Inconel 617 after being heat treated at 1093 K. (a) and (b) Surfaces exposed to molten LiF-22CaF₂ for 7913.8 h. (c) Molten salt/vapor interface after 400 h of exposure. (d) Weld exposed to LiF-22CaF₂ vapor for 7913.8 h. (a) Unetched. (b), (c), and (d) Etched

Table 4 Tensile properties of as-received Inconel 617 and Haynes alloy 230

Test temperature, K	0.02 % Yield stress		0.2 % Yield stress		UTS		Elongation	
	Average, MPa	Standard deviation, MPa	Average, MPa	Standard deviation, MPa	Average, MPa	Standard deviation, MPa	Average, %	Standard deviation, %
Inconel 617								
77	489.9	32.3	540.0	52.1	906.7	22.3	19.0	1.4
298	357.8	19.8	374.7	13.6	673.2	9.9	29.2	0.7
750	250.1	12.2	282.6	11.1	553.9	10.7	28.5	2.6
900	224.3	6.6	249.3	2.8	494.8	4.0	26.8	1.4
1050	215.1	3.9	239.8	3.1	417.3	5.6	25.6	0.2
1200	190.6	5.5	204.8	6.5	206.4	5.7	28.1	1.0
Haynes alloy 230								
77	678.7	2.9	734.3	20.2	1264.3	29.2	36.3	3.1
298	452.0	22.0	475.0	23.0	889.7	4.5	36.7	1.7
750	325.5	13.5	347.0	4.0	696.0	4.3	24.7	0.5
900	312.7	14.7	342.7	3.4	572.7	18.7	18.7	4.5
1050	308.3	20.0	335.3	7.7	460.3	17.0	7.3	1.9
1200	120.0	7.8	139.7	6.2	209.0	8.3	16.7	3.1

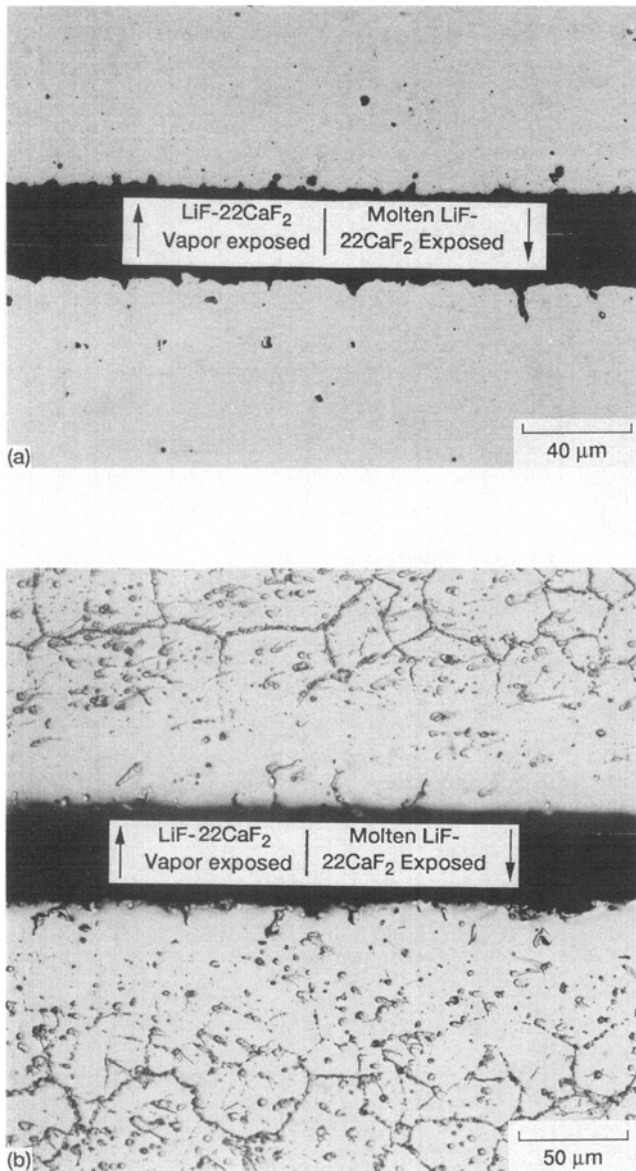


Fig. 4 Photomicrographs of Haynes alloy 230 in contact with molten LiF-22CaF₂ or its vapor after being heat treated for 10,000 h at 1093 K. (a) Unetched. (b) Etched

the worst case, the relative change for alloy 230 after any exposure is quite small (<2 mg/cm², Table 3).

Samples cut from the alloy 230 bread pan were utilized to assess damage metallurgically to the microstructure after the 10,000 h, 1093 K exposures. Alloy in contact with either the molten LiF-22CaF₂ salt or its vapor tended to have a rougher surface and wider surface-connected pits (Fig. 4a) than the starting material (Fig. 1a). The pits are basically remnants from the pickling process (Ref 10) given to the original sheet. Comparison of pit depths after exposure to those in the as-received alloy did not indicate any significant growth. Figure 4(b) illustrates the etched microstructure of alloy 230 after exposure, where a second-phase depletion zone about 45 μm thick exists beneath the molten-salt or salt-vapor exposed surfaces. Similar

regions were seen after 10,000 h vacuum exposure (Ref 12), and narrower bands were seen in the as-received alloy (~10 μm, Ref 10) or after 2500 h of exposure (~20 μm, Ref 10). After 10,000 h, 1093 K heat treatment in vacuum, the alloy 230 surface tends to be rougher than the as-received condition, and the surface-connected pits are wider and, perhaps, a bit deeper (Ref 12).

Scanning electron microscope and electron microprobe analysis of alloy 230 after 10,000 h exposure at 1093 K confirmed the existence of second-phase depletion zones. Based on carbon wavelength dispersive x-ray maps of interior regions, the particles that dissolved were carbides. Both the molten-salt and vacuum-exposed surfaces (Fig. 3b) had similar carbide deficient regions where essentially all grain boundary precipitates, as well as many of the intragranular particles, had gone into solution. In addition to the carbon loss in the second-phase depletion regions, chromium wavelength dispersive x-ray maps revealed that a small chromium gradient existed across the carbide depletion zones. Chromium-line scans revealed that the chromium content at the molten-salt and/or vacuum-exposed surfaces of alloy 230 was 1 to 2 wt% less than that in the interior.

Wavelength dispersive and energy dispersive analysis of the carbides in the interior of the sheet indicated that the large, round intragranular second phases (Fig. 3b) were tungsten-rich, chromium-poor carbides. Through x-ray dot maps, back-scattered electron images, and line scans, chromium was identified as the major metallic constituent in the grain boundary carbides. In addition, back-scattered electron techniques clearly demonstrated that the grain boundary carbides contained much less tungsten than did the intragranular carbides.

3.2 Tensile Properties

The 77 to 1200 K tensile properties of IN 617 after various periods of exposure to molten LiF-22CaF₂, vacuum, and the salt vapor at 1093 K are presented in Fig. 5 to 7, and listings of average values and their standard deviations are given in Tables 4 to 7. Similar results for alloy 230 after 10,000 h of exposure can be found in Fig. 8 and Table 4 and 8.

3.2.1 IN 617.

The 0.2 percent yield stresses, UTS values, and tensile elongations for IN 617 after being immersed in molten salt for various periods are shown and compared to the values for the as-received alloy in Fig. 5. With one exception, no major degradation of properties occurs as a result of exposure. Based on the existing data, it appears that 400 and 2500 h exposures can reduce the postexposure tensile ductility (Fig. 5c) between 77 and 900 K. While the residual ductilities are about half the as-received values at 77 and 298 K, much smaller relative reductions occur at 750 and 900 K. At the higher test temperatures (1050 and 1200 K), little degradation is seen; furthermore, after 7914 h of exposure to molten salt, the residual elongations at all tensile test temperatures are greater than those measured for the as-received alloy. Several other minor trends also exist; for example, the 77 through 750 K yield strengths (Fig. 5a) and the 77 K UTS (Fig. 5b) of molten LiF-22CaF₂ exposed IN 617 generally decrease with time of exposure. These degradations in strength are, however, at most 10%. Opposed to such losses in

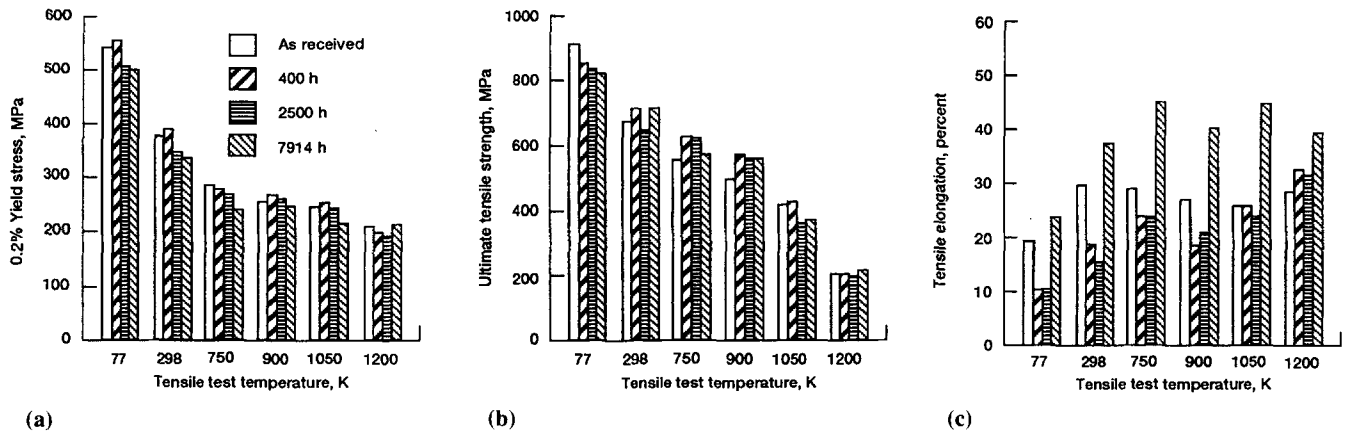


Fig. 5 Tensile properties of Inconel 617 after various periods of exposure to molten LiF-22CaF₂. (a) 0.2% yield strength. (b) Ultimate tensile strength. (c) Tensile elongation

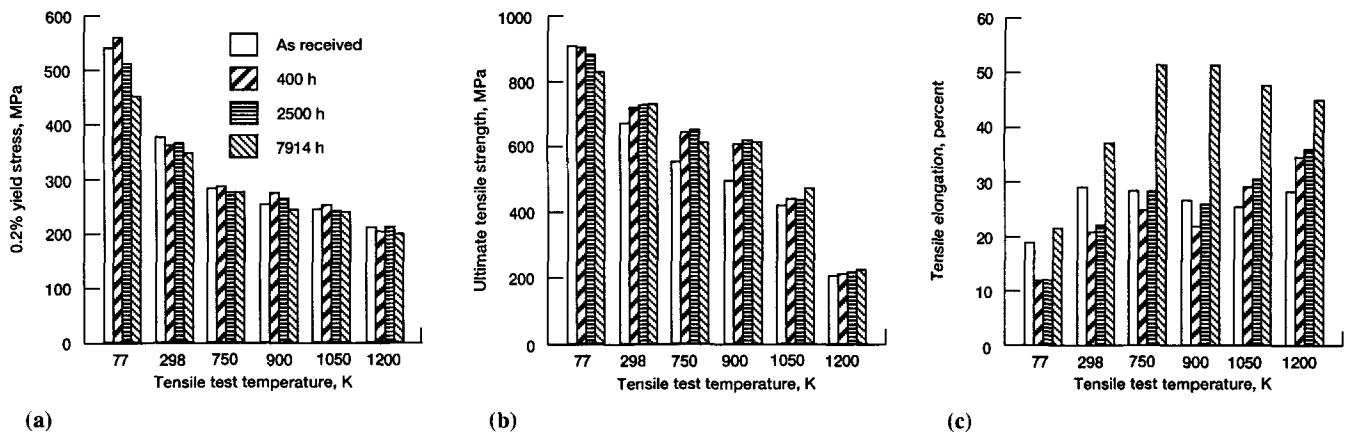


Fig. 6 Tensile properties of Inconel 617 after various periods of exposure to vacuum at 1093 K. (a) 0.2% yield strength. (b) Ultimate tensile strength. (c) Tensile elongation

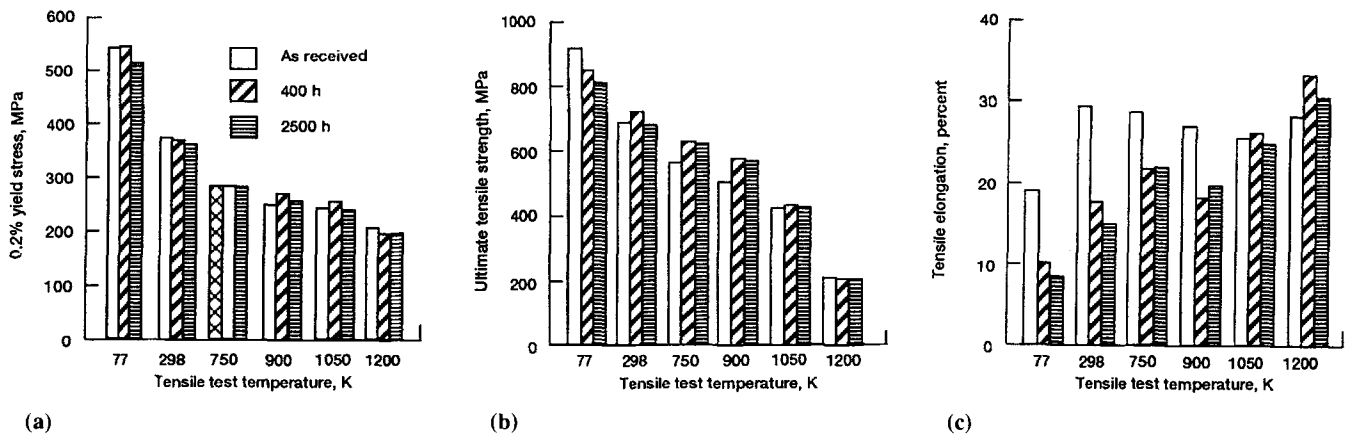


Fig. 7 Tensile properties of Inconel 617 after various periods of exposure to LiF-22CaF₂ vapor at 1093 K. (a) 0.2% yield strength. (b) Ultimate tensile strength. (c) Tensile elongation

strength, the postexposure 0.2% yield stresses at 900, 1050, and 1200 K (Fig. 5a) and the 298 to 1200 K UTS values equal or exceed the as-received properties.

The tensile properties of vacuum heat treated IN 617 (Fig. 6) follow basically the same strength and ductility trends demonstrated by the molten LiF-22CaF₂ exposed alloy (Fig. 5). In

Table 5 Tensile properties of Inconel 617 after exposures for 400 h at 1093 K

Test temperature, K	0.02 % Yield stress		0.2 % Yield stress		UTS		Elongation	
	Average, MPa	Standard deviation, MPa	Average, MPa	Standard deviation, MPa	Average, MPa	Standard deviation, MPa	Average, %	Standard deviation, %
Molten LiF-22CaF₂								
77	530.9	15.9	552.7	5.9	850.3	23.9	10.2	1.0
298	368.3	12.9	389.2	14.7	712.7	28.3	18.4	2.1
750	258.2	19.2	277.8	7.3	625.5	10.7	23.5	2.0
900	244.9	11.3	264.5	7.7	569.5	11.4	18.5	0.3
1050	232.0	8.7	249.1	5.6	427.2	5.1	25.7	1.2
1200	179.7	9.3	194.8	1.9	205.7	5.1	32.0	1.3
LiF-22CaF₂ vapor								
77	470.8	41.2	542.7	15.6	834.9	11.0	10.1	0.3
298	345.4	10.1	371.0	14.2	709.5	19.4	17.7	0.5
750	265.7	4.1	283.4	1.3	618.8	9.1	21.6	0.3
900	237.1	6.9	271.3	3.6	569.9	10.1	18.2	0.5
1050	236.1	4.0	251.2	4.0	428.2	4.4	25.9	0.2
1200	182.8	7.0	192.4	4.7	203.7	4.6	33.1	1.3
Vacuum								
77	495.2	27.9	557.0	10.5	904.8	20.0	12.1	1.2
298	335.4	8.6	361.6	6.5	718.2	19.5	21.0	1.6
750	271.3	3.7	285.5	4.2	643.3	20.5	24.9	1.2
900	249.7	4.4	271.3	1.3	605.2	17.2	21.8	1.7
1050	233.5	9.6	251.6	3.7	441.0	3.2	29.2	0.6
1200	181.3	8.7	198.6	1.6	211.6	2.5	34.5	1.0

Table 6 Tensile properties of Inconel 617 after exposures for 2500 h at 1093 K

Test temperature, K	0.02 % Yield stress		0.2 % Yield stress		UTS		Elongation	
	Average, MPa	Standard deviation, MPa	Average, MPa	Standard deviation, MPa	Average, MPa	Standard deviation, MPa	Average, %	Standard deviation, %
Molten LiF-22CaF₂								
77	459.9	55.7	504.4	27.7	836.7	5.1	10.4	0.5
298	321.7	12.6	346.6	15.6	646.3	62.9	15.3	3.8
750	250.2	4.4	265.2	1.0	622.1	10.9	23.4	1.3
900	246.5	6.0	255.4	0.9	558.1	17.5	20.4	1.0
1050	230.7	1.2	239.8	3.6	361.9	15.7	23.7	1.8
1200	185.2	2.8	189.1	0.9	198.0	1.6	31.2	0.7
LiF-22CaF₂ vapor								
77	446.7	13.7	512.2	6.6	798.5	12.6	8.5	0.3
298	326.7	6.6	362.9	4.4	665.5	9.8	14.9	0.8
750	253.2	7.6	280.5	10.3	610.4	6.3	21.9	0.6
900	249.2	1.2	256.7	1.5	558.8	9.2	19.7	1.2
1050	224.5	13.2	234.7	8.8	424.3	8.6	24.8	1.4
1200	175.2	8.9	192.5	1.4	202.2	0.6	30.2	0.5
Vacuum								
77	463.7	24.0	511.0	24.2	882.9	9.5	12.2	0.4
298	330.2	2.0	363.9	4.9	728.0	20.7	22.0	2.0
750	252.0	5.1	273.1	0.9	650.3	4.1	28.2	0.9
900	244.3	15.3	261.9	14.9	617.2	4.2	26.1	0.7
1050	230.6	2.6	238.8	0.8	431.6	10.7	30.6	0.9
1200	189.2	14.2	206.8	7.9	216.0	4.9	36.1	0.7

particular, 400 and 2500 h vacuum exposures reduce the 77 and 298 K tensile elongations by about one third in comparison to the as-received sheet. However, as opposed to molten-salt exposures, the 750 and 900 K tensile ductilities only degrade after the 400 h vacuum heat treatment; the 2500 h vacuum anneals

improve the 750 and 900 K elongations to the as-received levels. As for molten salt, the longest period of exposure produces enhanced ductility at all test tensile temperatures. Only the 77 K yield strength (Fig. 6b) and UTS (Fig. 6c) seem to be slightly degraded by increasing time of exposure. The tensile strength

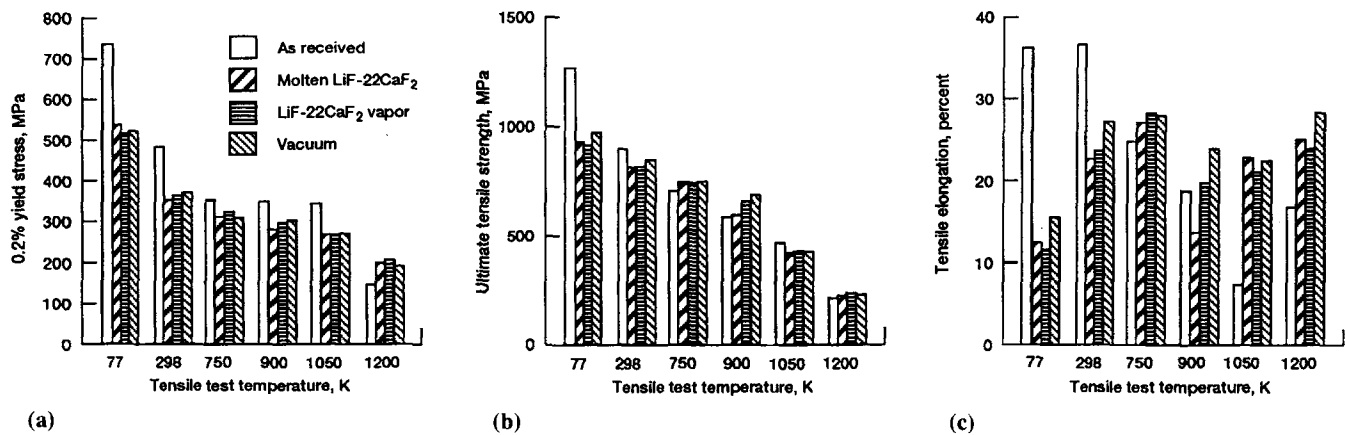


Fig. 8 Tensile properties of Haynes alloy 230 after 10,000 h of exposure to molten LiF-22CaF₂, its vapor, and vacuum at 1093 K. (a) 0.2% yield strength. (b) Ultimate tensile strength. (c) Tensile elongation

Table 7 Tensile properties of Inconel 617 after exposures for 7913.8 h at 1093 K

Test temperature, K	0.02 % Yield stress		0.2 % Yield stress		UTS		Elongation	
	Average, MPa	Standard deviation, MPa	Average, MPa	Standard deviation, MPa	Average, MPa	Standard deviation, MPa	Average, %	Standard deviation, %
Molten LiF-22CaF₂								
77	447.4	36.5	500.8	9.2	818.5	19.2	23.6	2.1
298	304.6	24.0	332.8	20.5	713.3	9.1	37.0	1.5
750	231.1	12.3	238.5	11.0	568.5	30.3	44.7	1.5
900	233.6	4.6	242.3	6.7	557.3	7.8	39.8	1.9
1050	206.0	6.4	210.2	6.9	371.8	42.6	44.1	1.0
1200	205.5	4.4	209.8	2.3	216.5	5.3	38.7	1.4
Vacuum								
77	390.9	43.1	452.9	21.9	829.0	8.0	21.7	0.9
298	307.3	41.2	346.4	28.0	728.1	14.5	37.3	3.3
750	263.1	10.2	272.5	11.9	612.2	48.5	51.3	2.9
900	236.1	4.9	241.7	5.8	613.0	8.0	51.3	2.4
1050	223.5	2.9	236.4	12.6	467.0	11.9	47.6	2.7
1200	182.2	32.6	194.9	29.3	222.9	11.3	45.1	3.5

characteristics between 298 and 1200 K either improve or remain unchanged after 400 to 7914 h vacuum heat treatments at 1093 K.

Due to a change of direction in the space station program, only the 400 and 2500 h LiF-22CaF₂ vapor-exposed IN 617 specimens were tensile tested, and the results are presented in Fig. 7. In general, the behavior is consistent with both the molten-salt (Fig. 5) and vacuum-exposed (Fig. 6) data for similar lengths of exposure. The 77 to 900 K tensile ductilities (Fig. 7c) are reduced in comparison to the as-received levels with the largest losses of about half at 77 and 298 K. All other property and test temperature combinations, with the exception of the 77 K UTS, show little degradation or dependence on time of exposure.

3.2.2 Alloy 230.

The average tensile properties of alloy 230 exposed for 10,000 h at 1093 K are presented in Fig. 8 as a function of the

various environments. Any prior exposure produces reductions in the 77 to 1050 K yield stresses (Fig. 8a) where at the two lower tensile test temperatures (77 and 298 K) fairly large degradations occur, about 30% from the as-received values. The 77 K UTS of alloy 230 also suffers an ~30% decrease from the as-received value after 10,000 h of exposure (Fig. 8b); however, the UTS values at higher test temperature reveal only minor reductions (298 and 1050 K) or slight increases (750, 900, and 1200 K). Large degradations in the low-temperature tensile elongations (Fig. 8c) take place after exposure to any environment. At 77 K, prior exposure to molten LiF-22CaF₂, its vapor, or vacuum produce about a two-thirds reduction in ductility; this property is likewise reduced by about one third at 298 K. Tensile elongations of exposed alloy 230, however, generally exhibit more ductility than the as-received sheet at the higher test temperatures. The only possible exception to this observation is the postexposure 900 K tensile ductility of material immersed in liquid salt (Fig. 8c). While the difference between exposed and as-received seems large, the standard deviation of

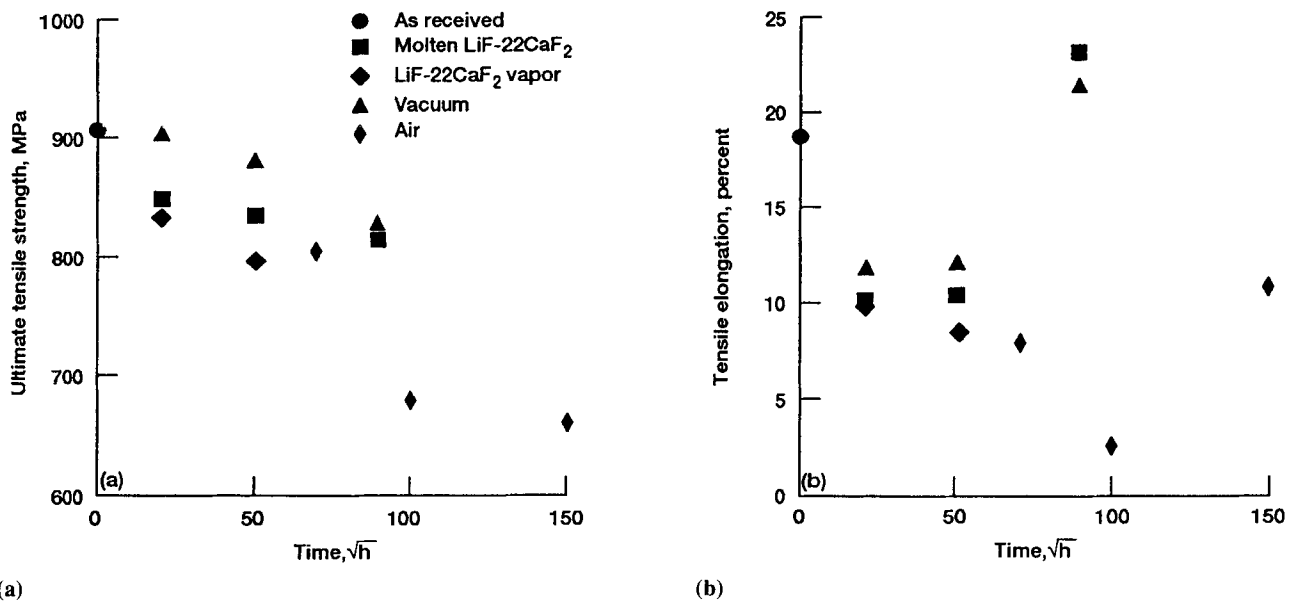


Fig. 9 The 77 K tensile properties of Inconel 617 after various periods of exposure to molten LiF-22CaF₂, its vapor, vacuum, and air at 1093 K. (a) Ultimate tensile strength. (b) Tensile elongation strength

Table 8 Tensile properties of Haynes alloy 230 after 10,000 h exposures at 1093 K

Test temperature, K	0.02 % Yield stress		0.2 % Yield stress		UTS		Elongation	
	Average, MPa	Standard deviation, MPa	Average, MPa	Standard deviation, MPa	Average, MPa	Standard deviation, MPa	Average, %	Standard deviation, %
Molten LiF-22CaF₂								
77	450.3	29.3	534.4	11.5	929.0	14.0	12.3	0.6
298	262.0	43.5	347.2	10.7	806.3	16.1	22.8	2.8
750	289.5	8.8	304.5	9.6	739.3	5.4	27.2	1.5
900	262.3	10.9	273.7	7.6	581.9	43.1	13.6	2.7
1050	247.1	3.3	262.2	6.2	404.2	6.6	23.0	1.6
1200	175.3	1.6	190.4	2.7	214.1	0.7	25.2	0.3
LiF-22CaF₂ vapor								
77	398.8	16.7	513.0	0.7	906.7	7.0	11.5	0.8
298	288.3	8.7	357.3	2.7	809.1	5.5	23.7	1.1
750	309.0	19.7	317.3	21.4	736.4	21.1	28.3	0.7
900	269.0	1.5	287.6	1.3	640.8	10.1	19.9	1.8
1050	244.6	4.8	258.2	2.8	416.3	17.6	21.2	0.7
1200	164.3	2.8	197.6	5.6	216.8	5.2	23.9	1.3
Vacuum								
77	406.4	10.1	516.9	5.7	968.1	17.8	15.4	0.9
298	291.7	19.7	366.0	3.7	839.3	7.5	27.2	0.9
750	285.3	1.0	301.0	2.5	739.9	24.7	28.0	1.5
900	278.3	10.1	294.9	16.1	669.8	44.9	24.1	0.6
1050	254.8	3.8	259.4	4.4	414.2	27.4	22.6	0.2
1200	169.1	2.2	183.3	4.4	204.7	4.8	28.4	2.4

4.5% for as-received sheet (Table 3) suggests that an average postexposure elongation of 13.6% is well within the 95% confidence limits for as-received alloy 230. Taken as a whole, the tensile property data in Fig. 8 (Table 8) do not reveal much evidence for the dependence of postexposure strength and ductility on environment. Thus the measured tensile characteristics were only affected by aging at 1093 K.

4. Discussion

The current results for IN 617 and alloy 230 indicate that the most prominent indicator of mechanical property degradation following exposures to molten LiF-22CaF₂, the salt vapor, and vacuum at 1093 K can be seen in the 77 K tensile data. Figures 9 and 10 present the compilations of the 77 K UTS values and

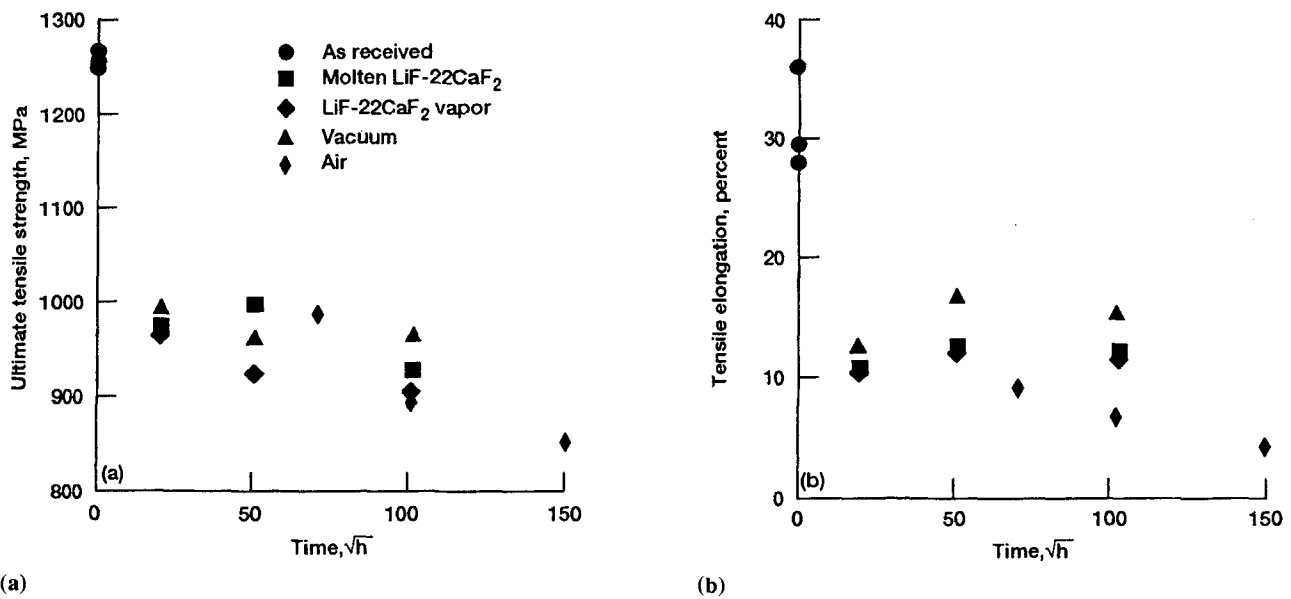


Fig. 10 The 77 K tensile properties of Haynes alloy 230 after various periods of exposure to molten LiF-22CaF₂, its vapor, vacuum, and air at 1093 K. (a) Ultimate tensile strength. (b) Tensile elongation strength

tensile elongations for both superalloys as functions of exposure environment and length of exposure where, for convenience, the square root of time at temperature is utilized as the abscissa. These figures include results from long-term 1093 K heat treatments in air (Ref 13) as well as shorter-term salt and/or vacuum exposures of alloy 230 (Ref 10).

4.1 IN 617.

Ultimate tensile strength of IN 617 at 77 K (Fig. 9a) reveals that with increasing time at temperature the UTS generally decreases. Through 8000 h, only minor losses are found ($\leq 10\%$), and there does not appear to be any real dependency of strength on the specific environment. However, longer exposures to air demonstrate an overall $\sim 30\%$ reduction in the 77 K UTS of IN 617 in comparison to the as-received value. Tensile elongations at 77 K (Fig. 9b) show a dramatic loss to $\sim 10\%$ (about half the as-received ductility) with any exposure lasting from 400 to ~ 5000 h. Longer times at temperature give a conflicting pattern. A time of 7914 h in either molten salt or vacuum leads to tensile elongations exceeding the as-received value, and after 22,500 h heat treatment in air, a ductility of $\sim 10\%$ was found. But 10,000 h exposure to air reduced the tensile elongation to 3.5%.

Summarizing the results in Fig. 3, 5, 6, 7, and 9 and Ref 13, it appears for exposures up to 8000 h long that 1093 K exposures of IN 617 to molten LiF-22CaF₂ or the salt vapor produce the same effects as a 1093 K heat treatment in vacuum or air. Therefore, no extra degradation in the tensile properties of IN 617 beyond that ascribed to simple thermal effects takes place from contact with the molten LiF-CaF₂ eutectic or its vapor at 1093 K. Longer 1093 K air exposure continuously reduces the strength properties of IN 617 (Ref 13); however, postexposure testing also reveals that the ductility after 22,500 h is greater than that after 4900 or 10,000 h. Because of this paradox and

the lack of tensile data after long-term 1093 K heat treatments in other atmospheres, it cannot be categorically stated that very long-term air exposures of IN 617 are equivalent to simple thermal annealing.

4.2 Alloy 230.

The 77 K tensile properties of alloy 230 are given in Fig. 10 after 1093 K exposure to air, vacuum, LiF-22CaF₂ vapor, and molten salt. Ultimate tensile strength (Fig. 10a) demonstrates a dramatic drop of $\sim 25\%$ after 400 h at temperature. Upon continued exposure, the UTS values slowly decrease to a point where after 22,500 h in air the 77 K UTS has lost another 5% compared to the as-received value. Ductility at 77 K (Fig. 10b) also shows a rapid decrease after a 400 h exposure to 1093 K; however, once reduced, the tensile elongation then basically remains at the same level or increases slightly for continued salt or vacuum exposures through 10,000 h. These strength and ductility results indicate that the salt does not produce any additional degradation beyond that due to simple thermal annealing, at least through 10,000 h. In comparison, the data from alloy 230 heat treated in air, however, suggest that air might be a detrimental environment. Prior air exposure consistently gives the lowest measured 77 K tensile ductility (Fig. 10b); thus, air heat treatments seem able to produce additional degradation beyond that of simple thermal annealing.

5. Summary of Results

Haynes alloy 230 and Inconel 617 were exposed to molten LiF-CaF₂, its vapor, and vacuum at 1093 K for periods up to 10,000 h to determine if salt exposure would attack these alloys. Examination of the postexposure microstructure and measurement of the 77 to 1200 K tensile properties revealed lit-

tle evidence for additional degradation beyond that due to simple thermal annealing.

Acknowledgment

The author acknowledges the review and helpful comments of Dr. Robert L. Dreshfield and the assistance of David Hull, Todd Leonhardt, and Jim Smith with their examination of the carbides and carbide-free zones in Haynes alloy 230.

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