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Type I and type II hot corrosion of Ni-based alloys without an incubation period

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Abstract.

A series of results obtained with different types of Ni-based alloys for turbine discs and blades, and exposed to various (Type I and Type II) hot-corrosion conditions (air, air + 150-400 ppm SO₂) is reported. Both the continuous mass-change measurements and the characterizations of alloys after a brief exposure at high temperature with sulphate deposits, in air or in air + SO₂ (g) atmospheres, clearly demonstrate fast oxidation rates from the earliest time of exposure in the presence of sulphate deposits, i.e. accelerated oxidation. In situ SEM observations also support these findings. It clearly appears that hot corrosion often starts with a direct reaction involving the metallic substrate, the gaseous species and the sulphate deposits, *i.e.*, that it occurs in most cases without any incubation period. The results can therefore be of interest for the development of complementary selection procedures for alloys and coatings

Main text.

Gas turbines (GT) components are subjected to severe thermal, mechanical, and chemical conditions. Chemical effects are generally due to oxygen (oxidation) and to the frequent presence of various deposits (hot corrosion). The latter results from pollutants in combustion gases and/or ingestion of sea salt, dust and volcano ashes [1,2], leading to the possible presence of alkali sulphates in deposits. These are responsible for an accelerated degradation phenomenon [3,4,5] of the nickel or cobalt based superalloys, often named hot corrosion. It usually involves a liquid phase, but cases where no liquid phase is expected have also been reported [6,7]. Hot corrosion of GT is a complex phenomenon, because of the multiplicity of interdependent

parameters (temperature, $O_2/H_2O/SO_2/SO_3$ partial pressures, chemical nature and amount of deposit, gas velocity, chemical nature of the alloys, etc.) and also of the variability of these parameters with time and operating geographical location. For this reason, several corrosion mechanisms [8,9,10,11,12,13] have been proposed, but papers dealing with the evaluation of hot corrosion rate and giving clear direction for the development of improved corrosion resistant alloys and coatings are scarce.

Furthermore, corrosion test results, typically involving a sodium sulphate deposit and a gaseous air + SO_2 flow that partially mimic the GT environment, are generally very difficult to compare, even when the test parameters appear to be similar. Many authors [1,9,14,15] agree on the fact that these phenomena are due to sulphate deposits and that the attack takes place in two stages. The first one is named incubation, and the second is propagation. During the incubation, the oxidation rate is assumed to be close to the oxidation rate in air without deposit. This period is depicted as the required time allowing the establishment of chemical conditions into the deposit such that a fluxing mechanism [9] of protective oxides layers can occur. For type II hot corrosion encountered around 600°C - 700°C , the period also includes the time required to form a molten phase [10,15,16] because sodium sulphate ($T_m = 884^\circ\text{C}$) is solid in this temperature range. Acidic fluxing in the gas phase [4] or induced by the alloy [17], basic fluxing [3,5] or synergistic fluxing [11] are suggested to be responsible of initiation (end of incubation) and to hinder the subsequent healing of a passive layer, leading to a rapid propagation stage. Propagation can be self-sustained under different flow conditions, depending on the solubility gradient of the oxides in the corrosion products [18,19,20]. For this stage, the reaction rate is reported to be very fast, the damage very local and in depth, affecting the alloys mechanical properties [21,22]. This is why the incubation time is generally examined when evaluating alloys, and a significantly longer incubation time is expected when developing a coating solution. This incubation period is generally defined for ordinary burner rig tests as the time elapsing before the first pitting is observed with the naked eye.

However, in a number of experiments that were carried out at lab scale by the present authors over the past years, the incubation period was often absent and the degradation was severe just after a few hours of exposure to the corrosive environment. These data and the variety of tested conditions show that the reactivity of materials in the presence of a deposit during the incubation stage needs to be further examined in order to develop new testing procedures for alloys and coatings selection. A few results showing this are presented hereafter.

The composition, in weight percent, of the tested Ni-based materials can be found in Table 1. All materials, with the exception of the model alloys (γ and γ' phases), were supplied by the SAFRAN group and were in the same metallurgical and heat treatment condition as expected for use in service. Details about their microstructure and manufacturing method can be found elsewhere [23, 24, 25, 26, 27, 28].

Table 1: Nominal or actual* compositions (in wt.%) of the tested alloys

%wt	Ni	Co	Cr	Al	Fe	Mo	Pt	Re	W	Ti	Ta	Hf	Nb	C	(ppm)
AD730	Bal.	9.0	16.0	2.0	4.3	3.0	-	-	2.5	3.6	-	-	1.1	-	500 (Zr)
R65	Bal.	13.0	16.0	2.1	0.9	4.0	-	-	4.0	3.7	-	-	0.7	-	160 (B) 500 (Zr)
γ *	Bal.	-	25.12	1.13	-	-	0.45	-	-	-	-	-	-	-	
γ' *	Bal.	-	4.0	10.91	-	-	0.49	-	-	-	-	-	-	-	
TROPEA*	Bal.	9.0	6.41	5.41	-	0.59	1.95	0.99	6.1	1.0	9.1	0.09	-	-	6.9 (S)
DS200-Hf	Bal.	9.5	8.6	4.90	0.02	-	-	-	11.8	1.9	-	1.58	0.86	0.1	150 (B) 100 (Zr)

Type II hot corrosion [13] occurs in a temperature range corresponding to that experienced by low-pressure turbine discs (LPTD) in service, *i.e.* 550°C-700°C. To assess the corrosion resistance, the salt spray method was used; further details can be found in [13,29]. The surfaces of three specimens of the nickel-base superalloy for LPTD AD730™ were ground to P800 grit, oxidized 2 h in lab air at 700°C, then sprayed with 1 mg.cm⁻² of Na₂SO₄ (T_m = 884°C). Samples were then hanged to a platinum wire (which serve as a catalyst for the oxidation of SO₂) and exposed at 650°C to air or air + 400 ppm SO₂ (g) flowing gas. Figures 1a and 1b present the measured mass change *vs* time and its derivate, respectively. In these tests, the mixture of air + 400 ppm SO₂ (g) was introduced from the beginning of the test for curve A; after 1 hour in air at 650°C for curve B and results are compared to those of a sample prepared with the same procedure, but exposed to air without SO₂ for curve C. At 650°C, the oxidation rate of this alloy in air (without SO₂) was extremely slow, even in the presence of the Na₂SO₄ deposit (curve C). Interestingly, for curve B, the rate of the reaction changed from pretty slow to a significantly higher rate at 1 hour, as soon as SO₂-containing air was introduced in the gas flow. The mass-change was linear from the start for curve A. Such a behaviour was not expected, since an incubation period, during which the mass change remained of the same order of magnitude as that obtained without deposit, was assumed to occur before reaching the conditions under which fluxing of protective oxides can progress [9,10,15,16,30].

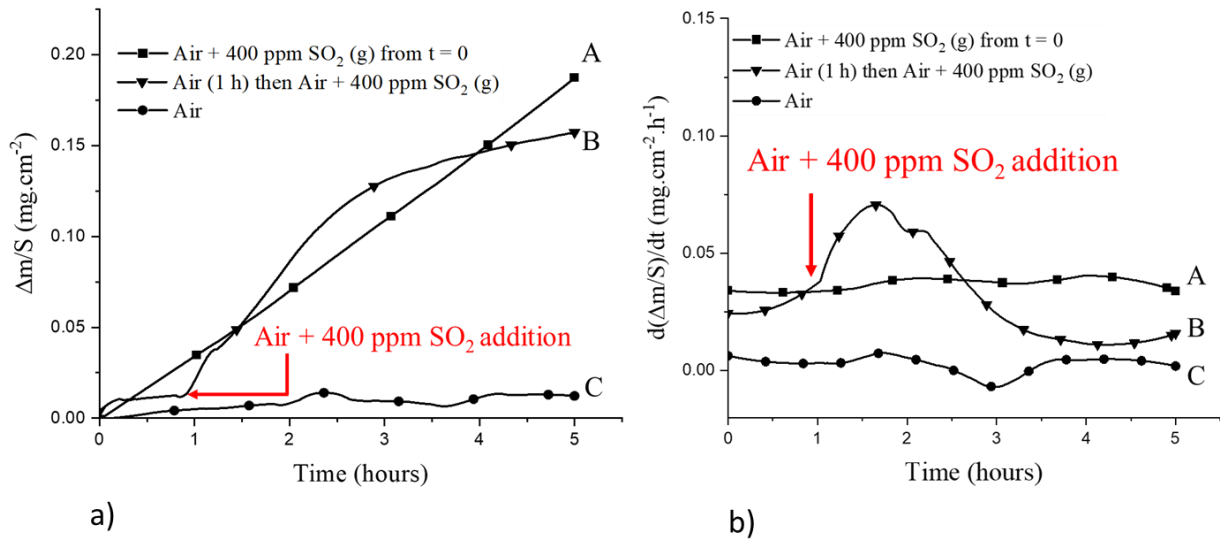


Fig.1: a) TGA (thermogravimetric analyzer) continuous measurements of mass change vs time and b) derivative of mass change vs time for AD730TM with 1 mg.cm⁻² Na₂SO₄ deposit exposed in air and/or in air + 400 ppm SO₂ at 650°C (the red arrow indicates the time at which SO₂ (g) is introduced for test B)

Figure 2 shows the cross-section of the nickel base superalloy R65, which is also dedicated to manufacture components such as LPTD. The samples were initially oxidized 2 h in lab air at 700°C, then 1 mg.cm⁻² deposit of the eutectic mixture Na₂SO₄-MgSO₄ (T_e = 670°C [31]) was applied prior to one-hour exposure of the sample to air + 150 ppm SO₂ (g) at 700°C. Severe local attacks were already present after this short time, scattered over the entire surface. The attack depth can locally reach 15-20 μm, which is pretty close to what was reported by Malacarne *et al.* [13] after 100 h of exposure in air + 400 ppm SO₂ (g) at 650°C in presence of a Na₂SO₄ deposit for AD730TM. The close order of magnitude between an exposure duration of 1 hour and another of 100 h in highly different conditions of gas and temperature was remarkable and raised the question of what had happened in Malacarne's study in the earliest hours of exposure.

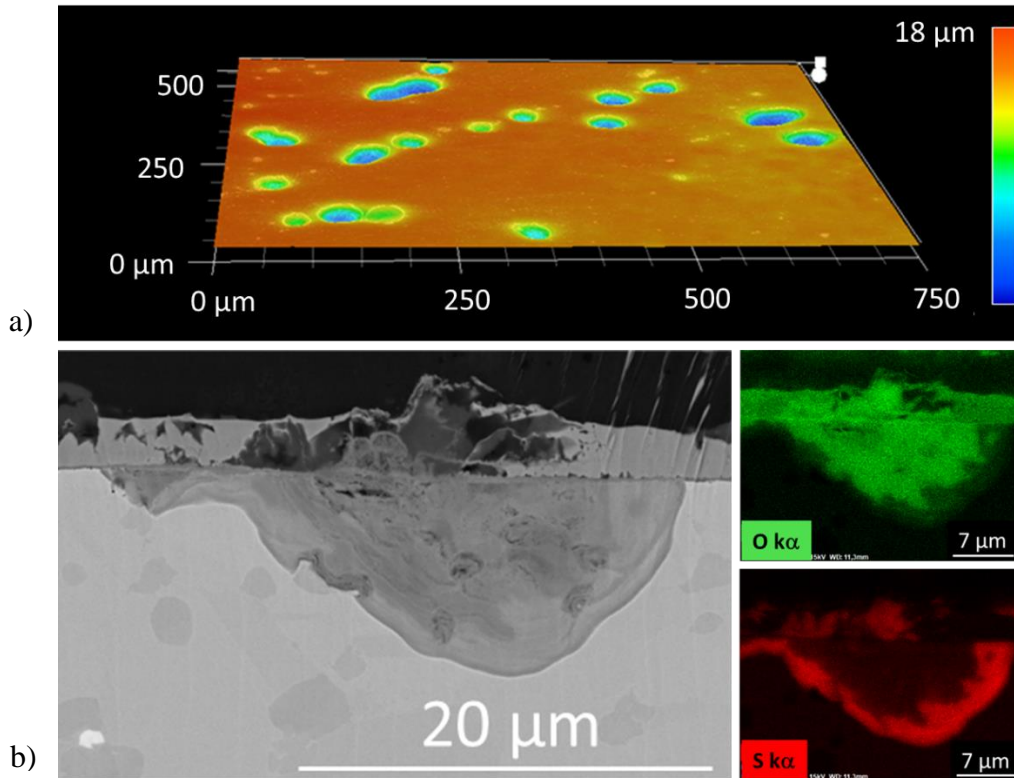


Fig.2: a) Sample surface topography and b) Backscattered image and X-ray elemental maps of the cross-section of R65 with $1 \text{ mg.cm}^{-2} \text{ Na}_2\text{SO}_4\text{-MgSO}_4$ deposit after just one hour of exposure in air + 150 ppm SO_2 at 700°C

Type I hot corrosion is found on aeronautical engines parts (such as high pressure turbine blades and nozzles) at higher temperatures (900°C - 1000°C), again due to the presence of sodium sulphate containing deposits. In this case, Na_2SO_4 is directly in a molten state. The Type I hot corrosion mechanism [4,9] also assumes an incubation period during which the protective oxide layer dissolves in the melt. In the literature [3,14], tests are generally carried out in air, but they can also be performed in an atmosphere simulating combustion gases (with the presence of sulfur oxides in the gas [6]). Both cases are examined hereafter.

Figure 3 presents the mass change recorded by TGA of two model alloys (γ and γ' ; see Table 1) manufactured to mimic each single phase of the γ/γ' -TROPEA nickel-based single-crystal superalloy. The tests were performed with a $1 \text{ mg.cm}^{-2} \text{ Na}_2\text{SO}_4$ deposit on bare samples, and the samples were exposed both to air and air + 400 ppm SO_2 (g) at 900°C . Mass variations of the samples without deposit and tested in air are also included in Figure 3. The data evidenced first that the mass gains did not evolve continuously with time of exposure. This was particularly true for the γ alloy in air with Na_2SO_4 deposit, for which the mass gain increased dramatically after about 9 hours and then stabilized. It can be noted that the oxidation rate of both γ and γ' samples in air enhanced from the very beginning of the experiments in presence of a deposit compared to the samples without any deposit. When SO_2 (g) was added to the gas, the oxidation rate changed. It tended to be linear with time for the γ' alloy, which confirmed the clear role of SO_2 (g) on the

oxidation rate [28]. This behavior again developed from the earliest minutes of the test; *i.e.*, without an incubation stage. For the γ alloy, the corrosion rate of the sample was not so dependent of the gaseous environment but remained higher than the oxidation rate with no deposit.

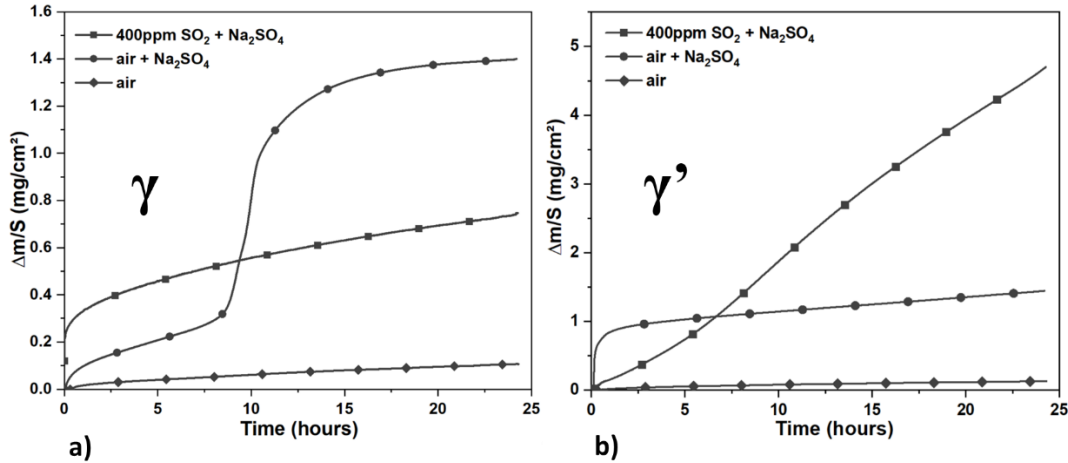


Fig.3: Mass change vs time of a) γ and b) γ' model alloys using thermogravimetry with $1 \text{ mg}\cdot\text{cm}^{-2} \text{ Na}_2\text{SO}_4$ deposit exposed in air + 400 ppm SO_2 (g) at 900°C

Cross-sections of both γ/γ' superalloys for blades, DS200+Hf and TROPEA, after 5 h at 700°C in air + 150 ppm SO_2 (g) and 3 h at 900°C in 150 ppm air (low vacuum in the SEM) are presented in Figures 4a and 4b, respectively. The DS200+Hf alloy was ground at P1200, oxidized 2 h in lab air at 700°C , before spraying $1 \text{ mg}\cdot\text{cm}^{-2} \text{ Na}_2\text{SO}_4$. For this alloy, the depth of the attack was significant for this short test duration; the affected zone reached 15-20 μm in depth. For TROPEA alloy, the surface was polished with 1200 to 4000 grade SiC papers, then with a diamond solution (1 μm) and with a 0.1 μm colloidal silica suspension (mirror polishing) prior to the hot corrosion experiments. Then, a few grains of sodium sulphate were sprinkled onto the as-prepared bare surface. The initial sulphate grains were less than 30 μm in size. Thus, considering the conditions used, the amount of sulphate salt was limited. For the TROPEA alloy, the experiment was performed in situ in an environmental scanning electron microscope (ESEM). Details of the procedure can be found in reference [32]. Once the target temperature (*i.e.* 900°C) was reached, the samples were held for an isothermal stage and images were recorded regularly at different magnifications to collect information at different scales. Frame times were adjusted according to the rate of the oxidation reaction. The movies that have been reconstructed after the experiments are included in the supplementary materials. The cross-section in Fig.4d corresponds to a small part of the affected zone of this sample after corrosion, which was far larger than the image size.

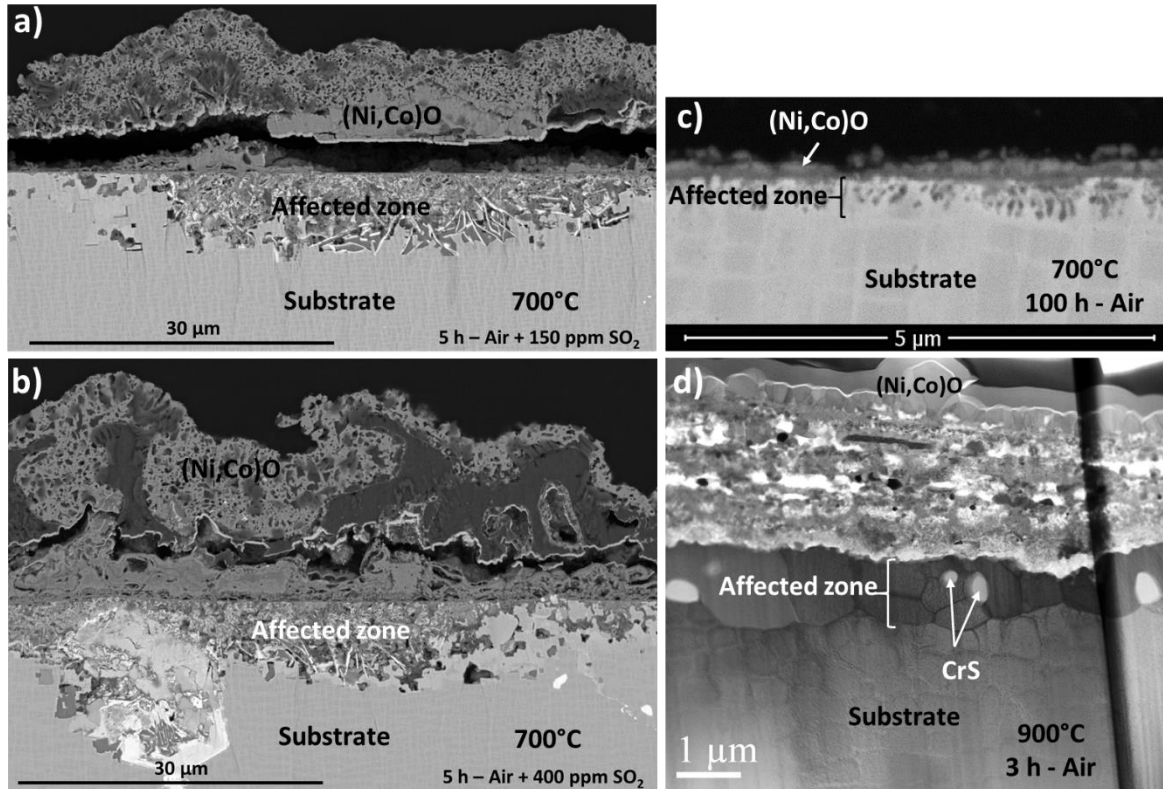


Fig.4: Cross-sections of DS200+Hf in presence of sodium sulphate after 5 h at 700°C in a) air + 150 ppm SO₂ ; b) air + 400 ppm SO₂ ; c) after 100 h oxidation in air at 700°C without deposit for comparison; d) TROPEA alloy exposed in 150 mbar air at 900°C for 3 h (the bright field STEM section corresponds to the sample for which the *in situ* observation is proposed in supplementary material)

The observations carried out on both γ/γ' superalloys clearly demonstrate significant microstructural changes in the alloy subsurface. These (Fig.4a and 4b) showed substantial evolution as compared to the ones obtained after oxidation in air, as illustrated by the cross-section of purely oxidized samples without deposit (Fig.4c) after 100 h of exposure in air. For both, DS200+Hf (Fig.4b) and TROPEA (Fig.4d), new γ grains replaced the original γ/γ' microstructure in the affected zone. Chromium-rich sulfides were also present in the affected zone of the TROPEA alloy (Fig.4d). Accurate TEM analyses did not reveal the presence of sodium in the corrosion products. Sodium sulphate appeared to be fully consumed after this short term *in situ* experiment. Concerning the experiments with DS200+Hf, the amount of sulphate was larger and so was the depth of the corrosion attack. This was really significant after 5 h in air + 150 ppm SO₂ (Fig.4b). All these results indicate that there was no need of long exposures to initiate a marked corrosion phenomenon. The alloys were already deeply affected after these short durations and their mechanical properties could be thus drastically lowered [22,33].

The cases presented, which cover the behaviour of a few nickel-based superalloys under conditions representative of Type I and Type II hot corrosion, show that attacks are already significant for extremely short durations. A number of authors have also observed fast mass change within a few hours [6,34,35]. Kofstad [34] or Jones [36], dealing with the determination of the corrosion rate at 700°C of pure nickel and CoCr alloys, both using thermogravimetry, mentioned that the corrosion rate can be very fast in the earliest minutes of the exposure in air + SO₂ (g). All our observations support that hot corrosion often starts with a direct reaction involving the metallic substrate, the gaseous species and the sulphate deposits, *i.e.* that it often occurs without any incubation period. These findings are critical for alloys and coatings development because the salt component – alloy reactions [37] as well as the corrosion products (oxides, sulfides, sulphates [38]), that develop during the earliest period of the interaction, deeply change the environmental thermodynamic conditions as well as the microstructure and the superficial composition of the alloys. But the in-depth attacks, that result of this rapid reaction, can also serve as a crack initiator leading to a severe lowering of mechanical properties such as fatigue strength [33]. Consequently, more attention should be paid to the impact of this short-term interaction developing future generations of alloys and coatings. Taking these short-term changes into account might enable on one hand the rationalization of hot corrosion test results, *e.g.* obtained using burner rig, and, on the other hand, should open the way to new testing methods for evaluating the integrity of components in hot corrosion conditions.

Supplementary materials

Video of the in situ observation of the corrosion of TROPEA at 900°C in air by Na₂SO₄ deposit

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Declaration of Conflicts of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Availability of data and material - The raw/processed data required to reproduce these findings can be shared on demand.

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