EXFOLIATION CORROSION RESISTANCE OF NEAR PEAK-AGED AA7449 AS A FUNCTION OF QUENCH SPEED AND AGEING TREATMENT

T.J. WARNER[1], R. DIF[1], P. LASSINCE[2], P. LEQUEU[2]
[1] Pechiney Centre de Recherches de Voreppe, B.P. 27, 38340 Voreppe, France
[2] Pechiney Rhenalu, ZI des Listes, BP 42, 63502 Issoire, France

ABSTRACT

The influence of variations in processing parameters on the sensitivity to exfoliation corrosion (EC) of 7x49 has been addressed in a programme of controlled quenches followed by a variety of near-peak ageing treatments on 38 mm plate and two gauges of extrusion. Detailed microstructural characterisation (optical microscopy and transmission electron microscopy) has been performed on selected product-quench-ageing combinations in order to study the critical microstructural features for resistance to EC of AA7x49. For these near-peak ageing treatments the following conclusions can be drawn: the EXCO performance is better for a product that has undergone a faster quench and a lower temperature ageing treatment; there is no simple relationship between sensitivity to exfoliation corrosion and electrical conductivity; for an identical quench and ageing treatment, the EXCO ratings can be ranked in order of grain structure alignment.

These observations are consistent with an underlying mechanism of EC depending on both the degree of alignment of the grain structure and the degree of solute segregation in the vicinity of the grain boundary. However the TEM observations demonstrate that there is no simple relationship between PFZ width or intergranular precipitate size and sensitivity to EC.

Keywords: Exfoliation corrosion, Plate, Extrusion, 7xxx alloys

1. INTRODUCTION

Airframe manufacturers increasingly require improved resistance to corrosion in very high strength aluminium alloys. In particular, there is a need for greater resistance to exfoliation corrosion (EC) of extrusion and plate destined for applications in which the compressive yield strength is the dominant requirement. In general, increased resistance to corrosion of such products can be achieved by overageing treatments [1, 2], with a corresponding strength penalty. However, in the case of extrusions in AA7349 and 7449, our initial results suggested that the industrial peak-aged temper (T6511) was at least as resistant to EC as slightly over-aged tempers. The goal of the work described in this paper was to confirm this result and to clarify the underlying metallurgical principles through a programme of controlled quenches and ageing treatments and concomitant microstructural characterisation of selected product-quench-ageing combinations.

2. EXPERIMENTAL

Samples were taken from industrial production of 7x49 at Pechiney Rhenalu's Issoire and Montreuil-Juigné plants from three different product forms: 38 mm 7449 plate, a thick gauge 7449 extrusion (22.5 mm wall thickness), and a thinner gauge 7349 extrusion (sampled from 4.7 mm thickness). All three products were sampled in the industrial T651(1) tempers.

Rectangular section bars of three different thicknesses (22 mm, 10 mm and 5 mm) were machined symmetrically about the mid-planes of the plate and the thick gauge extrusion. These bars were then re-solution heat treated, quenched by direct immersion in 20°C water, stretched 2%, and subjected to three laboratory near-peak ageing treatments (a slight under age at 120°C, a near-peak two step treatment (max temperature 150°C), and a slight overage (max temperature 150°C) designated respectively A, B and C). The thinner gauge extrusion was re-solution treated, quenched in water at 20°C, stretched 2%, and subjected to two near peak ageing treatments (near peak at 120°C, and an overage (max temperature 156°C), designated A and D respectively). In addition, the products were characterised after industrial T6 treatments.

Both the sensitivity to EC at the midplane of each bar (the original product's midplane) according to the EXCO accelerated test (ASTM G34) and the tensile properties were subsequently characterised.

An additional series of heat treatments on the industrially quenched thick-gauge extrusion was performed to investigate the influence of ageing temperature on EC sensitivity at a given strength. EXCO tests at 1/10 of the flange thickness and tensile tests were performed after ageing treatments at 120°C, 135°C, 140°C and 150°C, with heating ramps simulating an industrial furnace.

TEM observations were performed on twin-jet electropolished thin foils in a LEO 912 equipped with an omega filter.

3. RESULTS

3.1 Effect of quench speed and ageing treatment on exfoliation corrosion

The results of this part of the study are presented in table 1 and as figures 1 to 3. They can be summarised as follows:

- for a given ageing treatment the EXCO performance is better for faster quenches (thinner gauges, see e.g. figure 1);
- heat treatment B corresponds to the peak in both sensitivity to EC as assessed by the EXCO test and tensile yield strength for the thick gauge materials (see figures 2 and 3). Heat treatment A, which corresponds to a slight underage, is in general less sensitive to EC than the other two heat treatments;
- there is no simple relationship between sensitivity to EC and electrical conductivity. However, for the lab quenched samples a conductivity of 19-20 MS/m appears to correspond to peak sensitivity in the EXCO test;
- for an identical quench and ageing treatment, the EXCO ratings are better for the plate than for the thick gauge extrusion, which in turn are better than for the thin gauge extrusion.

Table 1. Tensile EXCO, and electrical conductivity data for the different heat treatments

Product	Quench type or bar thickness mm	Ageing treatment	Electrical conductivity MS/m	EXCO*	TYS MPa	UTS MPa	El %
7449 Thick gauge Extrusion	Industrial	T6511	18.1	EC	641	681	12
	22	A	17.9	EA/EB	643	684	11.7
	10		17.8	EA	651	693	11.4
	5		17.8	EA	649	696	12.6
	22	B	19.8	EC	650	675	11.3
	10		19.8	EB	654	678	11.3
	5		19.8	EB	655	682	10.9
	22	C	21.1	EC	633	661	11.2
	10		20.9	EB	632	659	12.1
	5		20.8	EB	623	662	12
7449 Plate	Industrial	T651	18.4	EA/EB	639	678	12.2
	22	A	18.3	EA	637	680	12.1
	10		18.3	EA	631	674	11.7
	5		18.6	EA	609	666	13.6
	22	B	20.3	EA/EB	643	667	11.4
	10		20.4	EA	632	659	9.7
	5		20.7	EA	602	657	10.8
	22	C	21.4	P/EA	618	647	10.9
	10		21.6	P/EA	611	641	12.1
	5		21.7	EA	602	640	10.7
7349 Thin gauge extrusion	Industrial	T6511	17	EA	730	762	9.9
	4.7	A	16.8	EA	677	717	10.6
	4.7	D	19.7	EC	630	667	9.4

* ASTM standard ratings except for split ratings (e.g. EA/EB) for which the inspector was unable to assign unambiguously only one of the two ratings quoted.

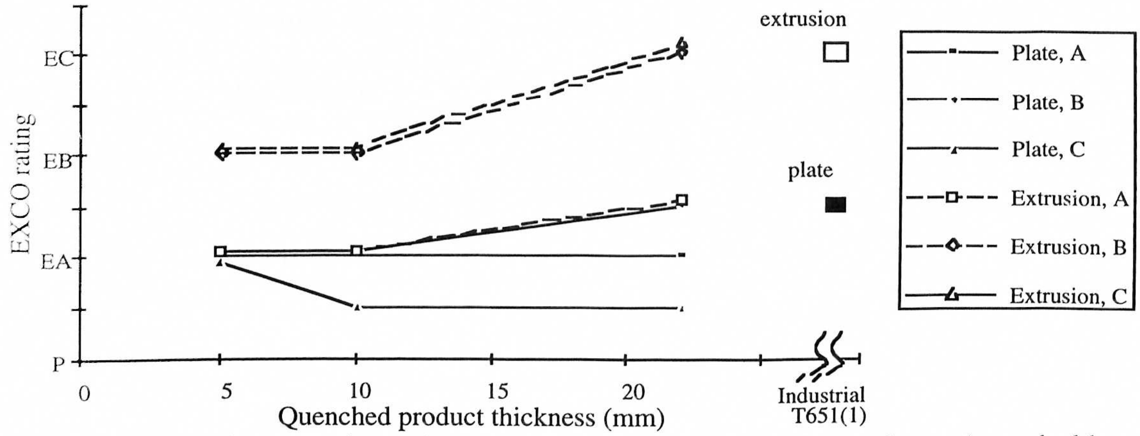

Figure 1. Exfoliation corrosion rating (EXCO test) as a function of quench rate (quenched bar thickness) for 7449 plate and thick-gauge extrusion after three ageing treatments (A, B, C).

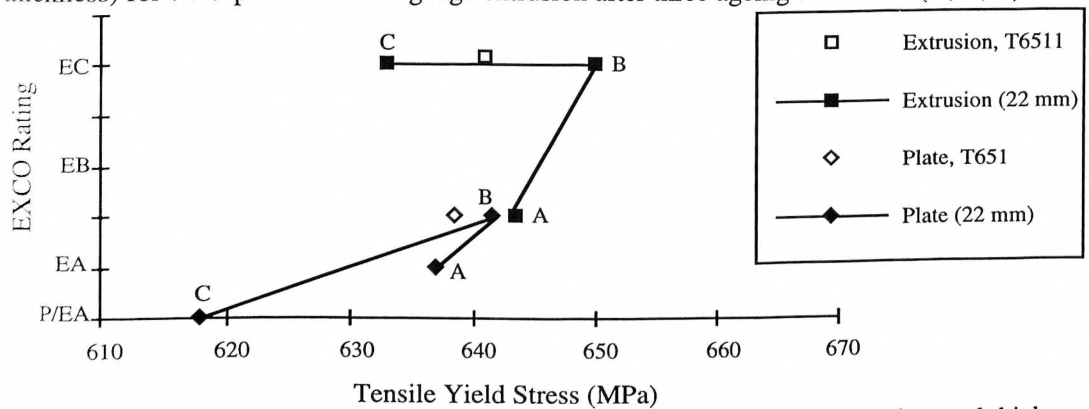

Figure 2. EC rating (EXCO test) as a function of tensile yield stress for 7449 plate and thick-gauge extrusion after an industrial quench or after a lab quench at 22 mm thickness.

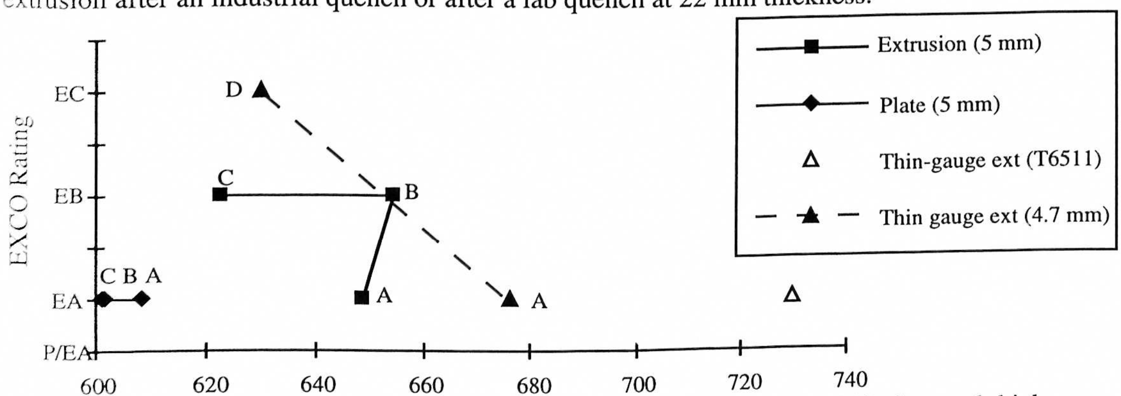

Figure 3. EC rating (EXCO test) as a function of tensile yield stress for 7449 plate and thick-gauge extrusion after a lab quench at 5 mm thickness, and for the thinner extrusion (thickness 4.7 mm) after an industrial quench or a lab re-quench for various ageing treatments.

3.2 Sensitivity to exfoliation corrosion as a function of ageing temperature

The sensitivity of industrially quenched thick gauge extrusion as a function of ageing temperature at peak yield strength is presented as figure 4. It is clear that sensitivity to EC at peak strength is increased by ageing the extrusion at a higher temperature. Moreover, the achievable peak strength is greater for lower ageing temperatures.

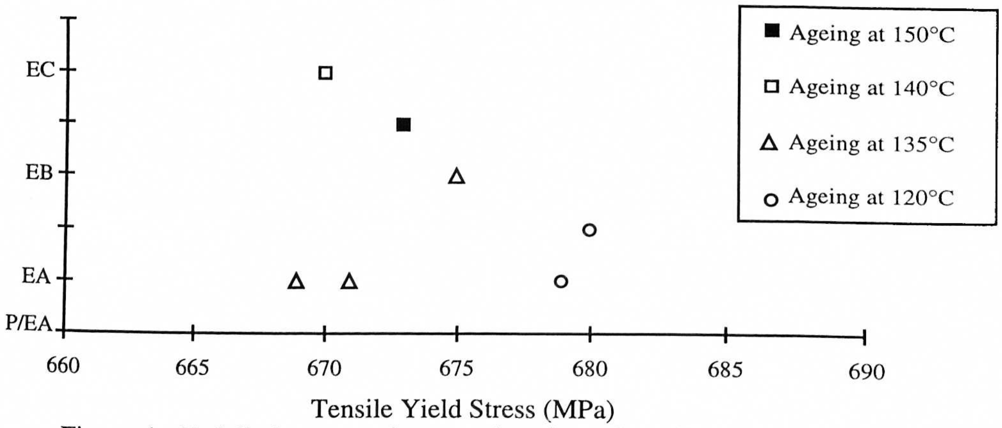

Figure 4. Exfoliation corrosion as a function of tensile yield stress for 7449 heavy gauge extrusion (sampled at th/10) aged to peak strength with ageing cycles with different peak temperatures. Note that the ageing treatment at 150°C included a first step age at 120°C.

3.3 Microstructural characterisation

Typical optical micrographs after a chromic etch to reveal grain / sub-grain boundaries are presented as figure 5. The plate has a partially recrystallized microstructure, whereas both the extruded products are unrecrystallized, the thicker-gauge product showing evidence of recovery.

Characteristic TEM micrographs of the near grain boundary areas corresponding to some of the combinations of quench speed and ageing treatment are presented as figure 6. Quantified parameters obtained from image analysis of a number of micrographs are presented as table 2. From figure 6, it can be observed that the grain boundary microstructures are all similar. However the following significant differences can be observed:

- the density of grain boundary precipitation (not measured owing to the complexity of the measurement of foil thickness) is higher for the slower quenched (thicker gauge) materials;
- faster quenching and the lower temperature ageing treatment give smaller PFZs;
- for identical quench rate, the intergranular precipitate size is greater for the higher temperature ageing treatment.

Table 2. Grain boundary microstructural parameters

Product	Thick gauge extrusion						Thin gauge extrusion		Plate	
Quench	Industrial		Lab (22 mm)		Lab (22 mm)		Industrial		Industrial	
Artificial age	T6511		A		C		T6511		T651	
Intergranular precipitate dimensions										
	Large	Small	Large	Small	Large	Small	Large	Small	Large	Small
Number of meas.	35	35	45	45	32	32	39	50	46	46
Mean (nm)	28.8	12.6	36.7	13.6	56.2	21.6	38.4	12.6	28.0	13.2
95% confidence interval (nm)	3.7	1.5	4.4	1.0	7.2	1.6	5.8	1.7	3.0	1.1
Precipitate free zone widths										
Number of meas.	7		11		10		12		11	
Mean (nm)	13.9		14.9		17.5		10.7		14.8	
95% confidence interval (nm)	1.6		0.8		1.6		0.7		0.9	

4. DISCUSSION

It is generally recognised [1, 2] that sensitivity to EC of copper-rich 7xxx alloys results from the conjunction of an aligned grain structure and sensitivity to intergranular corrosion (IGC). Sensitivity to IGC itself is a purely electrochemical phenomenon [3, 4] determined by the existence of a microgalvanic couple between a grain boundary feature constituting a continuous anodic path with respect to a cathodic matrix (e.g. a continuous film of Al_3Mg_2 precipitation in Al-Mg alloys or a copper-depleted zone near the grain boundary for Al-Cu alloys).

The results presented above can thus be rationalised through consideration of these two factors. Thus, for a given combination of quench and heat treatment, the ranking of EXCO ratings is related to the degree of alignment of the grain structure: the thin-gauge extrusions (most aligned) being most sensitive, the plate (partially recrystallized and lower aspect ratio grains) least. For these near peak-aged copper rich 7xxx alloys, sensitivity to IGC depends largely on the extent to which the heat treatment cycle has permitted the development of concentration profiles in the vicinity of the grain boundary [5], and in particular copper depletion [6]. The subsequent desensitisation with further over-ageing, associated with reduced matrix solute content and the corresponding equalisation of both solute concentrations and solution potentials [3], has apparently not been reached for the extruded products after the near-peak ageing treatments employed in this study. However, although the peak strength appears to be reached for approximately the same ageing treatment for the two 7449 products, desensitisation to EC appears to occur at a lesser overage for the plate. There appears thus to be some influence of the grain structure (degree of recovery/recrystallization ?) on the kinetics of (de)sensitisation to EC.

On the macroscopic level, the observed reduction in sensitivity with faster quenches (which limit heterogeneous precipitation during quench) and lower ageing temperatures (which favour homogeneous precipitation over heterogeneous precipitation [7]) can be rationalised in terms of reduced micro-segregation of solute in the vicinity of the grain boundaries. However, the microstructural observations demonstrate that there is no simple relationship between PFZ width or intergranular precipitate size and the results of the EXCO test. The TEM data do however confirm that the intergranular precipitates after a higher temperature heat treatment are coarser (see comparison of heat treatments A and C in table 2). Quantification of grain boundary areal fraction and/or the concentration profile in the vicinity of the grain boundary would probably help to clarify the relationship between microstructure and exfoliation corrosion sensitivity.

5. CONCLUSIONS

For these near-peak ageing treatments the following conclusions can be drawn:

- EXCO performance is better for a product that has undergone a faster quench and a lower temperature ageing treatment;
- there is no simple relationship between sensitivity to EC and electrical conductivity;
- for an identical quench and ageing treatment, the EXCO ratings can be ranked in order of grain structure alignment.

These observations are consistent with an underlying mechanism of EC depending on both the degree of alignment of the grain structure and the degree of solute segregation in the vicinity of the grain boundary. However the TEM observations demonstrate that there is no simple relationship between PFZ width or intergranular precipitate size and sensitivity to EC. There appears to be an (unquantified) relationship between the quantity of intergranular precipitation and sensitivity to the EXCO test. Seacoast exposures of some of the above-mentioned products are currently in progress to confirm the trends observed in the accelerated tests.

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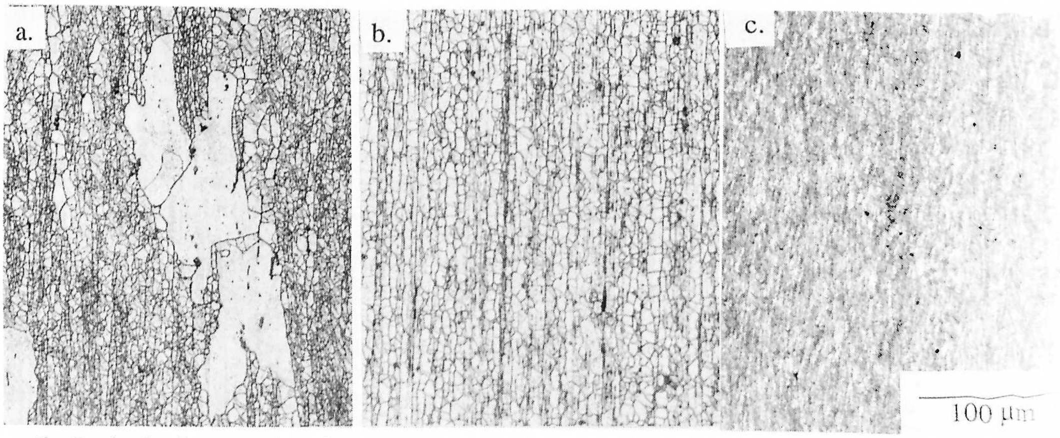

Figure 5. Optical micrographs after a chromic etch showing characteristic grain structures in the ST plane of (a) the 38 mm plate, (b) the thick-gauge extrusion, and (c) the thin gauge extrusion.

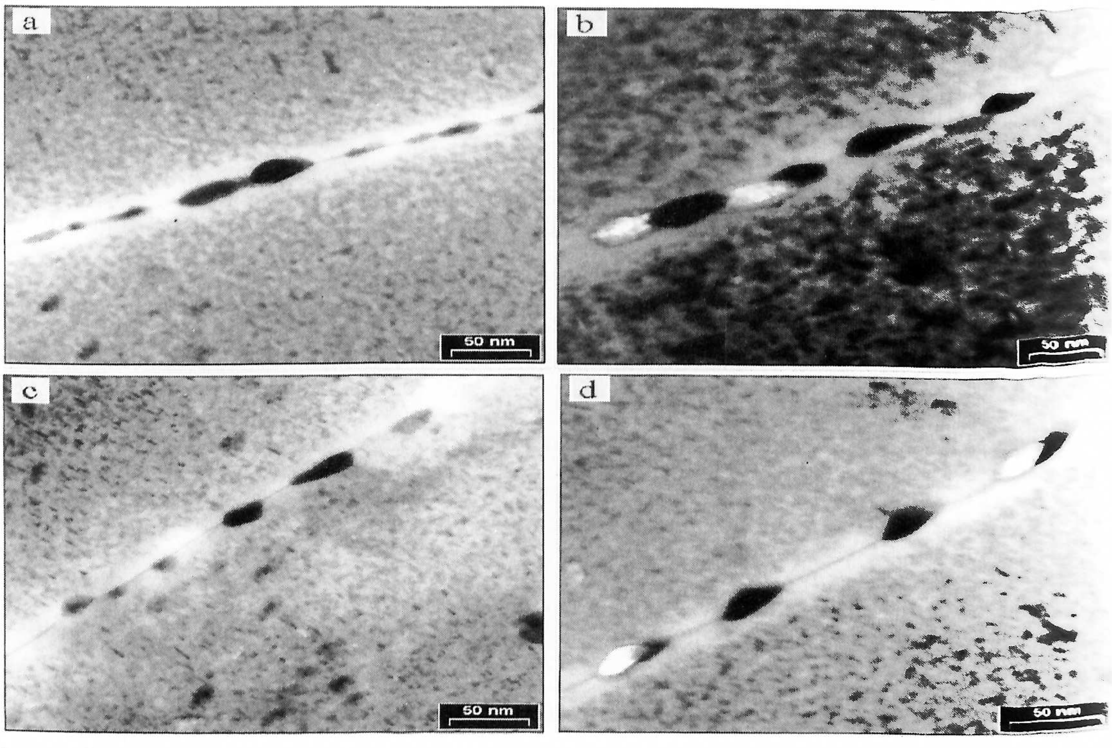

Figure 6. Bright field TEM micrographs showing characteristic near grain boundary microstructures for (a) thick-gauge 7449-T6511 extrusion, (b) thick-gauge 7449 extrusion after ... mm quench and ageing C, (c) 7449-T651 plate, (d) 7449-T6511 thin-gauge extrusion.