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CORRELATION BETWEEN ABRASION TESTS (FOLLOWING BS 6161 PART 18) AND DIFFERENT ANODISING CONDITIONS

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INTRODUCTION

An anodic oxidation coating consists of aluminium oxide built up by chemical conversion of the surface of the aluminium substrate. The anodic coating has the well-known microcellular structure of fine pores and its density can change through the thickness, being the surface layer more porous and softer than the inner layer, close to the metal interface. The average density depends on the anodising conditions.

Electrolyte temperature represents the most critical factor for the production of good quality oxide layers. Agitation of the electrolyte is fundamental to remove the heat generated at the surface of the aluminium work piece during the anodising process. High operating temperatures result in a partial dissolution of outer layers of as-formed anodic coating, making it soft and more porous.

Sulphuric acid concentration turns out to be critical only at high anodising temperatures: the higher is the acid concentration the lower is the anodising voltage required, but this fact leads to greater drag-out and higher acid consumption.

With the aim to evaluate the influence of the main process parameters on the abrasion resistance of oxide layers, a large number of specimens was prepared varying one at a time each of these parameters.

This paper deals with the abrasion tests, particularly the “manual abrasion test” according to BS6161 part18, carried out on the oxide layers used in the field of decorative or architectural applications.

EXPERIMENTAL PROCEDURE

Abrasion testing have been carried out in accordance to the current standard practices, considering the “manual abrasion test” (known as “Clark Test”) and the “Taber abrasion test”. Moreover, a certain emphasis was given to the results obtainable by using a new developed test apparatus, based on the “Clark Test” procedure.

Besides the variables of the anodising process, the possible effects of the subsequent oxide colouring process have been also considered. In fact, oxide colouring can be obtained through different methods that can change its structure (as it happens in the interference colouring process).



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Then comparative results were discussed and correlated to the different anodising and colouring conditions.

QUALANOD – EURAS/EWAA Quality Label Specifications

QUALANOD Specifications concerning quality requirements of the anodic oxidation coatings on wrought aluminium for architectural purposes relate mainly to the DC process in electrolytes of sulphuric acid or sulphuric/oxalic acid, and integral colour processes. Referring to sulphuric acid electrolytes, typical anodising conditions are so defined:

- sulphuric acid concentration not more than 200 g/l;
- aluminium content not more than 20 g/l;
- chloride content not more than 100 mg/l.
- bath temperature not above 20°C (for external application);
- anodic oxidation thickness of class 15 up to class 25 (for external application);
- average current density of 1.5 A/dm² up to 2,0 A/dm².

Sulphuric anodising process produces a transparent coating, having very good corrosion resistance. Aluminium alloys commonly used in architectural structures, after anodising by this process, have a natural silver grey appearance. The oxide cellular-like structure enables also organic/inorganic colour dyes to be absorbed into the pores of the coating.

Specimen preparation

Aluminium substrate: EN AW-5005 cold rolled aluminium alloy /temper H14

Nominal size of the test panels: 160 x 125 mm

Pretreatment: degreasing in an alkaline solution

Anodising: in sulphuric acid electrolyte (200 g/l or 300 g/l H₂SO₄), at a temperature of 15 – 20 – 25 – 30 – 35°C and an anodic current density of 2 – 4 – 8 – 16 – 32 A/dm², always using a good agitation of the bath (improved by means of air flow).

For comparison, at the temperature of 20°C, some test panels were also anodised without air flow.

Colouring: - tin-based electrolytic colouring (conventional composition with acid content 20 g/l H₂SO₄)

- interference colouring (preliminary treatment in sulphuric acid solution + electrolytic colouring).

Sealing: cold sealing based on nickel fluoride, followed by ageing by hot water treatment, as recommended by QUALANOD.

Specimens of suitable dimensions were carefully cut from each sample to be tested. They were identified with the following numerical mark:

- a) **2** (=200 g/l H₂SO₄) or **3** (=300 g/l H₂SO₄)
- b) bath temperature
- c) current density
- d) **1** = blue-grey interference colouring (12min in sulphuric acid electrolyte + 2 min in electrolytic colouring) or
2 = black electrolytic colouring (8min) or



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- 3 = medium-bronze electrolytic colouring (2min) or
4 = uncoloured oxide coating

Manual Abrasion Test (according to BS 6161 part18)

This test method is used as product quality control (but, at the same time, also as process capacity control) to evaluate the surface quality of an anodic oxidation coating, simply determining whether or not the coating is harder than a specified abrasive paper. The test is carried out manually, pressing an abrasive strip (a glass or a garnet coated paper 12 mm wide, the latter for hard anodised coating) against the test area. A good structure of anodic oxidation coating will be harder than the abrasive paper, which skids on the surface showing no deposit or very little white powder on it.

Two procedures are considered:

- Method I : after 10 double strokes, with an amplitude of 25 mm to 30 mm, no deposit or very little white powder must be detected on the abrasive paper;
- Method II : after 50 double strokes, refreshing the abrasive paper at every 10 double strokes, a loss of thickness no more than 2 µm must be measured.

Both method tests were performed on all the samples, producing the abraded track perpendicularly to the rolling direction of the base material (probably the most severe condition), since no indications are given by BS Standard in respect to the abrading direction. In preliminary tests no meaningful differences were found between the results obtained along the two principal directions of the base material.

The test results are reported in *Tab. 1*.

It is necessary to notice that Manual Abrasion Test is susceptible of quite scattered results when the anodic coating has a poor quality surface; in this case, we demonstrated that two operators within the same laboratory are likely to obtain differences in “loss of thickness” up to 2 µm (depending on the load applied by each operator) and consequently they can give either a positive or a negative result. Only for good quality anodic coatings no differences between two operators were found, since the abrasive action doesn't appear whichever is the load applied on the surface. For these reason the Manual Abrasion Test can be properly used in qualitative determinations.

Instrumented Abrasion Test (based on BS6161 part18)

Since the standardized manual method provides a numerical criteria to establish the quality level of the anodic oxidation coating, having nevertheless ascertained that this method is not able to give an accurate result, we repeated some tests introducing a load control device. In this way we could investigate about the range of results obtainable on the same sample when specific different loads are applied against the anodic oxidation surface; in other words, we tried to quantify the reproducibility of the test performed by different operators in different laboratories.

The new developed test method was set up by QUALITAL, starting from the manual test method described in BS 6161 part 18 and changing it into an instrumented test. The mechanical apparatus is shown in *Fig. 1*. The test piece is fixed on the moving table, a



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strip of glass coated paper (Grade 00) is wrapped round a block of a pencil eraser, held firmly in the lower part of the column capable of carrying weights up to a mass of 4 kg. At every 10 double strokes, the abrasive paper is refreshed and the loose powder is removed from the test area.

This test apparatus allows to apply a constant and specified load, exactly perpendicular to the surface, producing an uniform abrasion track on the specimen (*Fig. 2*).

Instrumented Abrasion Test results are reported in *Tab. 1*, as “loss of thickness”, together with Manual Abrasion Test results. Referring to the relative rating values, the two methods generally agree but, the single values of the Manual Test can be hardly related to corresponding values of Instrumented Test for only one load. Surely, Instrumented Test allows to obtain an improved repeatability precision of results.

The bar graph of *Fig. 3* shows a comparison between the results obtained on Colour 1 (the most critical sample) and Colour 3 (the least critical sample). Generally, increasing the load applied on the coating surface also the “loss of thickness” value increases; when a load increase does not cause a further “loss of thickness” it means that the reached oxide layer is harder than the abrasive paper.

The diagrams of *Fig. 4a*) and *4b*) summarize all Instrumented Abrasion Test results, respectively for 200 g/l and 300 g/l sulphuric acid solutions; in particular, they give the correlation “anodising temperature vs. current density” as limit operating conditions which are able to satisfy the requirement of a “loss of thickness” not more than 2µm.

Taber abrasion test

Taber abrasion test is usually performed to qualify “Hard anodic films of great thickness”, according to the Standards ISO 10074 and UNI 7796. In this case of anodic coatings for architectural purposes, Taber tests were restricted to 5000 revolutions; some tests, that were continued up to 10000 revolutions (as required for hard anodic coatings), have not given additional information.

The Taber test requires a 100 mm square and flat specimen, with a central hole so that it can be placed on the revolving turntable of the equipment. Two abrasive wheels CS-17 were used, each one applying 1000g load onto the rotating specimen. The relative motion of specimen and abrading wheels forms a 10 mm wide circular track, whose total area is about 25 cm², and resulting abrasion marks are crossed arcs (*Fig. 5*).

In the test program, both the most and the least critical samples (as detected in the previous abrasion tests) were considered; that is, respectively Colour 1 and Colour 3 samples.

The specimens were weighed to the nearest tenth of a milligram before the beginning of each test and again after a specified number of cycles (1000-3000-5000), previous 10 minutes stay into a silica-gel dryer. The results are reported in *Tab. 2* as “average loss of mass” (in mg) of two tests performed on both the surfaces of the same sample. Single values determined on the same sample were found to differ between them ± 0,5 mg per 1000 revolutions.

At the same cycle intervals (1000-3000-5000), the loss of coating thickness was also determined by using an eddy current meter, representing an alternative characteristic to confirm the abrasion resistance of the samples. More precisely, the “average loss of



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thickness” was considered, calculated from the thickness measurements taken in eight points evenly distributed along the abraded track. Also these values are reported in *Tab. 2*.

In comparing the wear resistance of similar materials, the “Taber wear index” is often considered. It is the loss in mass (mg) per thousand cycles of abrasion: the lower the wear index, the better the abrasion resistance of the surface material. The values so obtained are only representative of the used set of test conditions. Taber wear index were calculated for 1000 – 3000 – 5000 revolutions of the specimen (*Tab. 2*).

The bar graphs of *Fig. 6* show a quite good correlation between “loss of mass” and “loss of thickness”, so they both can be considered to really represent the abrasion resistance of the experimented anodising conditions. Accuracy of results mainly depends on correct weighing of the specimen, taking care to maintain constant room temperature and relative humidity during the test program. As an example, 12°C less than the normal temperature (at 50% relative humidity) was found to produce decreases of abrasion rate up to 3 mg per 1000 revolutions when the most critical samples were tested.

RESULTS

Depending on the anodising conditions and the colouring finishes, the consistency of the oxide coating can vary and abrasion testing is a useful method to find out the intrinsic quality of the layer.

All the performed tests have shown that the maximum abrasion resistance is achieved when anodising temperature is 20°C in 200 g/l sulphuric acid concentration.

Taking into consideration the various process parameters, the global results have pointed out the following trends (*Tab. 3*):

Sulphuric acid concentration

An increase in electrolyte concentration has a detrimental effect on abrasive wear resistance of the anodic coating, under the same remaining conditions. Only Taber Test does not clearly put this effect in evidence.

Current density

An increase in current density always produces an improvement of abrasion resistance, under the same remaining conditions. This effect is clearly shown by all the test method experimented.

Anodising temperature

An increase in bath temperature always produces a detrimental effect on abrasion resistance of the anodic coating, under the same remaining conditions. This effect is clearly shown by all the test method experimented.

Bath agitation



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Samples anodised at 20°C without air flow agitation of the bath, generally show a lower abrasion resistance. Only Taber test does not point out such a different behaviour.

Sample colour

The test results put in evidence that Colour 3 generally shows a better wear resistance than Colour 1. For this reason they were chosen to perform the complete test program and compare the three kinds of abrasion tests.

The obtained results confirm that the medium-bronze electrolytic coloured sample has the higher abrasion resistance (due to the shortest colouring time), while the blue-grey interference coloured sample has always the worse performance (due to the further anodising treatment in sulphuric acid electrolyte, in order to produce pore enlargement).

CONCLUSION

Abrasion resistance can be related to the quality of the anodic oxidation coating which is greatly dependent upon the conditions of anodising and colouring.

Manual and Instrumented Abrasion Tests are able to give clear information about possible differences in product quality, while this does not always happen in the case of Taber Test (see *Tab. 3: H₂SO₄ Solution and Bath agitation parameters*).

Manual or Instrumented Abrasion Tests, giving the same ranking results of the Taber Test, are surely more convenient and less expensive than the latter because they take a short time to be performed and the test area can be a very small flat surface, located on a non-critical part of test piece.

Furthermore, the results obtained by means of Instrumented Test machine (in terms of “loss of thickness”) are more accurate and show a better repeatability than those obtainable by hand.

Then the Instrumented Abrasion Test can be very useful to the assessment of the anodised coating quality, having previously defined the specific test load to be applied on the basis of the product requirements.

ACKNOWLEDGMENTS

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- ISO 10074:1994, “Specification for hard anodic oxidation coatings on aluminium and its alloys”, Annex B – TABER abrasion test method
- UNI 7796:1997, “Anodic oxidation coatings on aluminium and aluminium alloys – Hard anodic films of great thickness – Requirements and general instructions of test”, Appendix B – TABER abrasion test method



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Tab. 1 – Results of Manual and Instrumented Abrasion Tests vs. anodising and colouring conditions

Anodising Process Parameters					Manual Test		Instrumented Test		
Sulphuric solution (g/l)	Anodic temp. (°C)	Current density (A/dm ²)	Colour (*)	Layer thickness (µm)	Powder Meth 1 (**)	Δ thick Meth 2 (µm)	Δ thickness Meth 2 (µm)		
							2000 g applied	3000 g applied	4000 g applied
200	15	2	1	22	no	0			
200	15	2	2	19	no	-1			
200	15	2	3	19	no	0			
200	15	2	4	22	no	0			
200	15	4	1	21	no	0			
200	15	4	2	22	no	0			
200	15	4	3	18	no	0			
200	15	4	4	17	no	0			
200	20 s.a. (***)	2	1	17	+++	-4	-6	-6	-6
200	20 s.a.	2	2	17	+++	-3	0	-1	-5
200	20 s.a.	2	3	16	+	0	0	0	0
200	20 s.a.	2	4	16	++	-1	0	-1	-4
200	20 s.a.	4	1	17	++	-3	-5	-7	-8
200	20 s.a.	4	2	16	+	1	0	0	0
200	20 s.a.	4	3	18	+	-1	0	-1	-1
200	20 s.a.	4	4	20	+	-2	-4	-6	-6
200	20 s.a.	8	1	18	+	-1			
200	20 s.a.	8	2	18	+	-1			
200	20 s.a.	8	3	22	++	-3			
200	20 s.a.	8	4	19	+	-1			
200	20	2	1	19	no	0	0	0	0
200	20	2	2	19	no	0	0	0	0
200	20	2	3	18	no	0	0	0	0
200	20	2	4	18	no	0	0	0	0
200	20	4	1	19	no	0	0	0	0
200	20	4	2	18	no	0	0	0	0
200	20	4	3	17	no	0	0	0	0
200	20	4	4	20	no	0	0	0	0
200	20	8	1	20	+	0			
200	20	8	2	18	no	0			
200	20	8	3	17	no	0			
200	20	8	4	17	no	0			
200	20	16	4	17	no	0			
200	25	2	1	18	+++	-7	-4	-7	-9
200	25	2	2	18	+++	-6	-2	-5	-6



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Anodising Process Parameters					Manual Test		Instrumented Test		
Sulphuric solution (g/l)	Anodic temp. (°C)	Current density (A/dm ²)	Colour (*)	Layer thickness (µm)	Powder Meth 1 (**)	Δ thick Meth 2 (µm)	Δ thickness Meth 2 (µm)		
							2000 g applied	3000 g applied	4000 g applied
200	25	2	3	18	++	-3	-3	-5	-6
200	25	2	4	17	++	-4	-1	-3	-4
200	25	4	1	18	+	-1	-1	-1	-2
200	25	4	2	20	+	0	0	0	0
200	25	4	3	19	+	0	0	0	0
200	25	4	4	18	no	0	0	0	0
200	25	8	1	19	no	0			
200	25	8	2	23	no	0			
200	25	8	3	17	no	0			
200	25	8	4	17	no	0			
200	25	16	1	20	no	0			
200	25	16	2	23	+	0			
200	25	16	3	26	+	0			
200	25	16	4	17	no	0			
200	30	4	1	17	+++	-4	-3	-5	-5
200	30	4	2	17	+++	-6	-5	-6	7
200	30	4	3	17	++	-5	-3	-5	-5
200	30	4	4	19	+++	-4	-4	-7	-8
200	30	8	1	21	++	-2			
200	30	8	2	18	+	-1			
200	30	8	3	21	++	-1			
200	30	8	4	19	+	-1			
200	30	16	1	16	+	0			
200	30	16	2	21	++	-1			
200	30	16	3	16	++	0			
200	30	16	4	18	+	0			
200	30	24	4	25	+++	-2			
300	20	2	1	18	++	-1	-3	-5	-5
300	20	2	2	19	++	-2	-4	-6	-6
300	20	2	3	19	+	-1	0	0	0
300	20	2	4	19	+	-1	-1	-1	-1
300	20	4	1	19	no	0	-1	-1	-1
300	20	4	2	21	no	0	-1	-1	-1
300	20	4	3	22	no	-1	-1	-1	-1
300	20	4	4	22	no	-1	-1	-1	-1
300	20	8	2	18	no	0			
300	20	8	4	21	no	0			
300	20	16	2	21	no	0			



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Anodising Process Parameters					Manual Test		Instrumented Test		
Sulphuric solution (g/l)	Anodic temp. (°C)	Current density (A/dm ²)	Colour (*)	Layer thickness (µm)	Powder Meth 1 (**)	Δ thick Meth 2 (µm)	Δ thickness Meth 2 (µm)		
							2000 g applied	3000 g applied	4000 g applied
300	20	16	4	21	no	0			
300	20	32	4	22	no	0			
300	25	4	1	20	+++	-2	-2	-3	-3
300	25	4	2	21	++	-1	-1	-3	-4
300	25	4	3	20	+++	-1	-1	-4	-5
300	25	4	4	20	++	-2	-2	-3	-4
300	25	8	1	20	+	-2			
300	25	8	2	19	+	-1			
300	25	8	3	20	+	0			
300	25	8	4	20	++	-1			
300	25	16	2	19	no	0			
300	25	16	4	21	no	0			
300	25	32	1	21	+	0			
300	25	32	2	18	++	-1			
300	25	32	4	20	+	0			
300	30	8	1	20	+++	-3			
300	30	8	2	20	+++	-4			
300	30	8	3	20	+	-3			
300	30	8	4	21	++	-3			
300	30	16	1	17	+	0			
300	30	16	2	22	+	-1			
300	30	16	3	19	+	-1			
300	30	16	4	19	++	-4			
300	30	32	1	18	+	0			
300	30	32	2	18	+	0			
300	30	32	4	19	+	-1			
300	35	16	2	18	+	-3			
300	35	16	4	20	++	-2			
300	35	32	2	17	+	-2			
300	35	32	4	19	++	-1			

(*) Identification mark

- 1 = Blue-grey interference colour
- 2 = Black electrolytic colour
- 3 = Medium bronze electrolytic colour
- 4 = uncoloured oxide coating

(**) Chalky powder observation

- no = no deposit
- + = slight deposit
- ++ = steady deposit
- +++ = dense deposit

(***) s.a. = without air flow



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Tab. 2 – Results of Taber Abrasion Test

Sample	Loss of mass (mg)			Loss of thickness (µm)			TABER wear index (mg / 1000 rev)		
	1000 rev	3000 rev	5000 rev	1000 rev	3000 rev	5000 rev	1000 rev	3000 rev	5000 rev
2.20s.a.2.1	10,4	18,6	29,2	1	3	5	10,4	6,2	5,8
2.20s.a.2.3	5,3	11,6	16,9	1	2	3	5,3	3,9	3,4
2.20s.a.4.1	8,2	24,0	35,1	2	5	7	8,2	8,0	7,0
2.20s.a.4.3	7,1	15,8	23,5	1	3	4	7,1	5,3	4,7
2.20.2.1	8,0	19,4	27,2	1	4	5	8	6,5	5,4
2.20.2.3	5,5	14,2	24,7	1	2	4	5,5	4,7	4,9
2.20.4.1	4,6	14,4	24,0	1	3	4	4,6	4,8	4,8
2.20.4.3	6,4	14,3	19,8	1	2	3	6,4	4,8	4,0
2.25.4.1	9,3	21,6	46,8	2	4	9	9,3	7,2	9,4
2.25.4.3	9,9	22,9	28,5	1	3	4	9,9	7,6	5,7
3.20.4.1	2,8	10,8	24,8	1	2	5	2,8	3,6	5,0
3.20.4.3	7,1	17,6	40,9	1	3	7	7,1	5,9	8,2
3.25.4.1	4,8	21,8	32,5	2	5	7	4,8	7,3	6,5
3.25.4.3	9,5	31,7	39,7	2	6	7	9,5	10,6	7,9



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Tab. 3 – Influence of anodising process parameters on the results of the tree kind of abrasion tests

Process Parameters	Manual Abrasion Test	Instrumented Abrasion Test	Taber Test
Sample Colour	In the almost totality of the experimented anodising conditions, Colour 1 and Colour 3 samples are respectively the most and the least critical to the abrasion resistance, even in comparison with the uncoloured one	The results have shown the same ranking of abrasion resistance among the samples, with loads applied of 2000 - 3000 g	Colour 1 sample is less resistant than Colour 3 only with H ₂ SO ₄ solution 200g/l (on the contrary, with 300g/l solution)
H₂SO₄ Solution	Under the same remaining conditions, a drop of abrasion resistance is seen to happen if H ₂ SO ₄ concentration is increased from 200 to 300 g/l	Under the same remaining conditions, a drop of abrasion resistance is seen to happen if H ₂ SO ₄ concentration is increased from 200 to 300 g/l	The influence of H ₂ SO ₄ concentration is not so evident. Increasing H ₂ SO ₄ concentration, Colour 1 sample is seen to improve abrasion resistance (the contrary happens to Colour 3 sample)
Bath agitation	The samples anodised without air flow agitation of the bath, are seen to have a lower abrasion resistance (experimented conditions: 20°C and current density of 2 - 4 - 8 A/dm ²)	The samples anodised without air flow agitation of the bath, are seen to have a lower abrasion resistance (experimented conditions: 20°C and current density of 2 - 4 - 8 A/dm ²)	The samples anodised without air flow agitation of the bath, are seen to have a lower abrasion resistance for samples with 4 A/dm ² current density, while no substantial differences are seen for samples with 2 A/dm ² current density.



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Current density	Under the same remaining conditions, an improvement of abrasion resistance is seen to happen by increasing the current density	Under the same remaining conditions, an improvement of abrasion resistance is seen to happen by increasing the current density	Under the same remaining conditions, an improvement of abrasion resistance is seen to happen by increasing the current density
Bath temperature	Under the same remaining conditions, a worsening of abrasion resistance is seen to happen to all the samples by increasing the bath temperature	Under the same remaining conditions, a worsening of abrasion resistance is seen to happen by increasing the bath temperature	Under the same remaining conditions, a worsening of abrasion resistance is seen to happen by increasing the bath temperature



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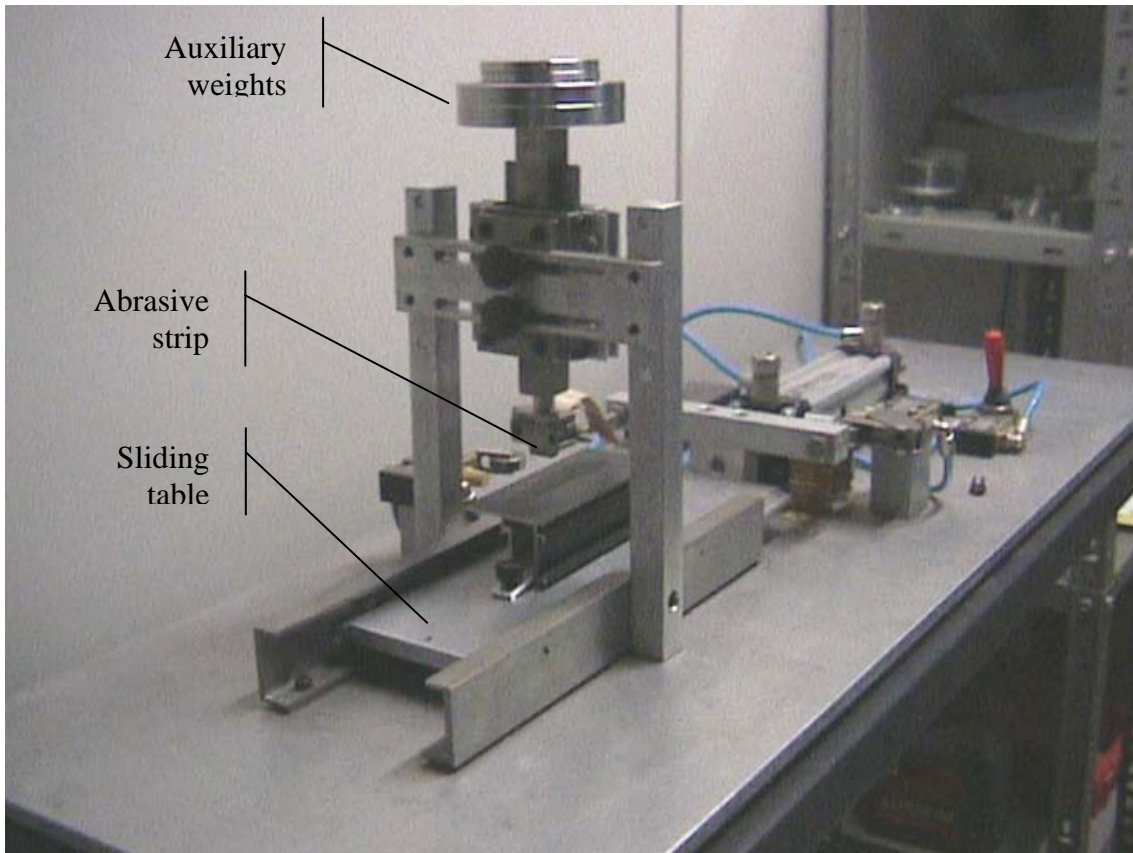
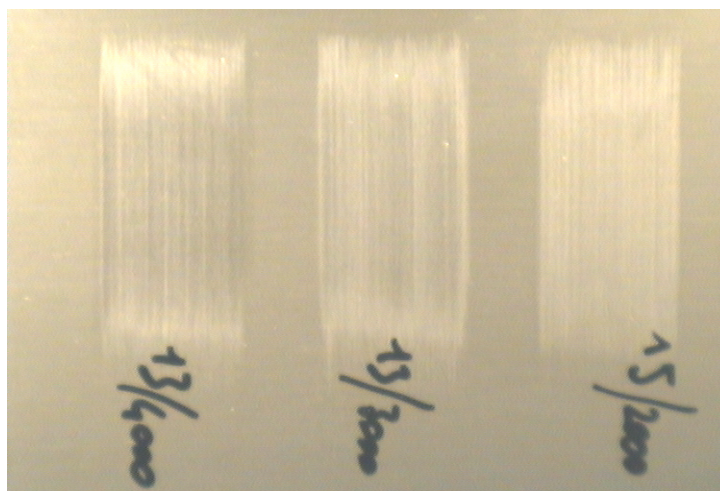


Fig. 1 – Instrumented Abrasive Test machine, set up for testing an anodised extruded profile.





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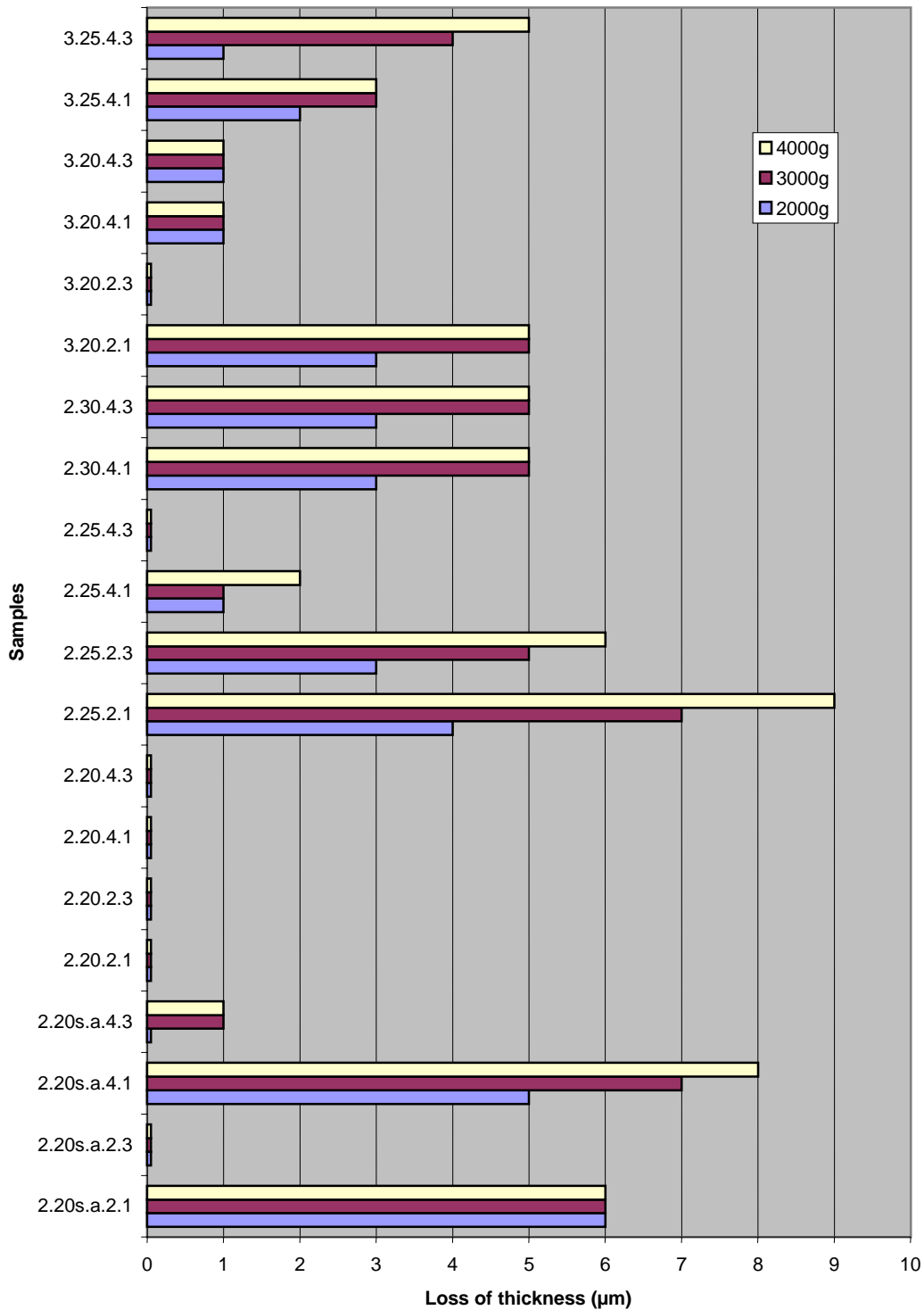
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Fig.2 – Abrasion tracks obtained on an anodised sample by using “Instrumented test machine” and applying different loads (2000 – 3000 – 4000g) for 50 double strokes





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Fig. 3 – Comparison of the Instrumented Abrasion Test results obtained on Colour 1 and Colour 3 samples



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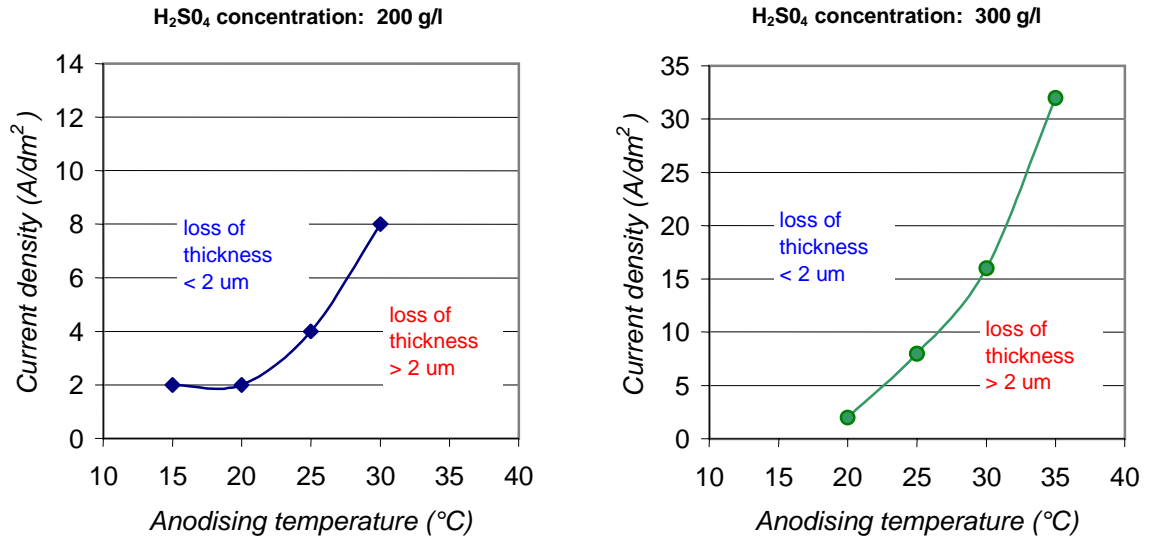
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a)

b)

Fig. 4 – Limit operating conditions of anodising process on the basis of Instrumented Abrasion Test results

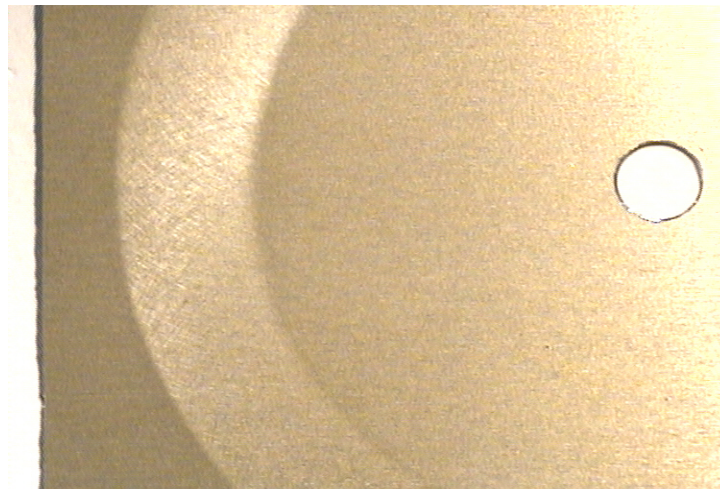


Fig 5 – Partial view of the abrasion track obtained on an anodised sample by using Taber test machine and applying a load of 1000g for 5000 revolutions.



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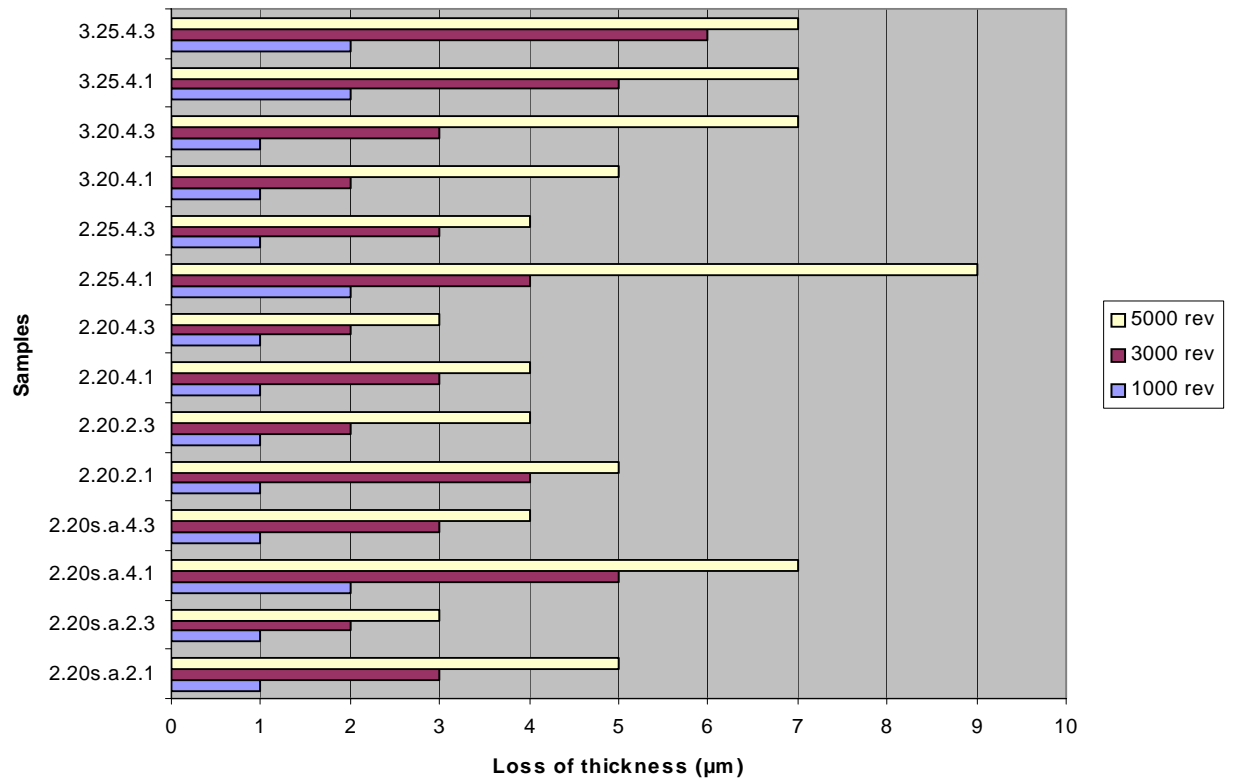
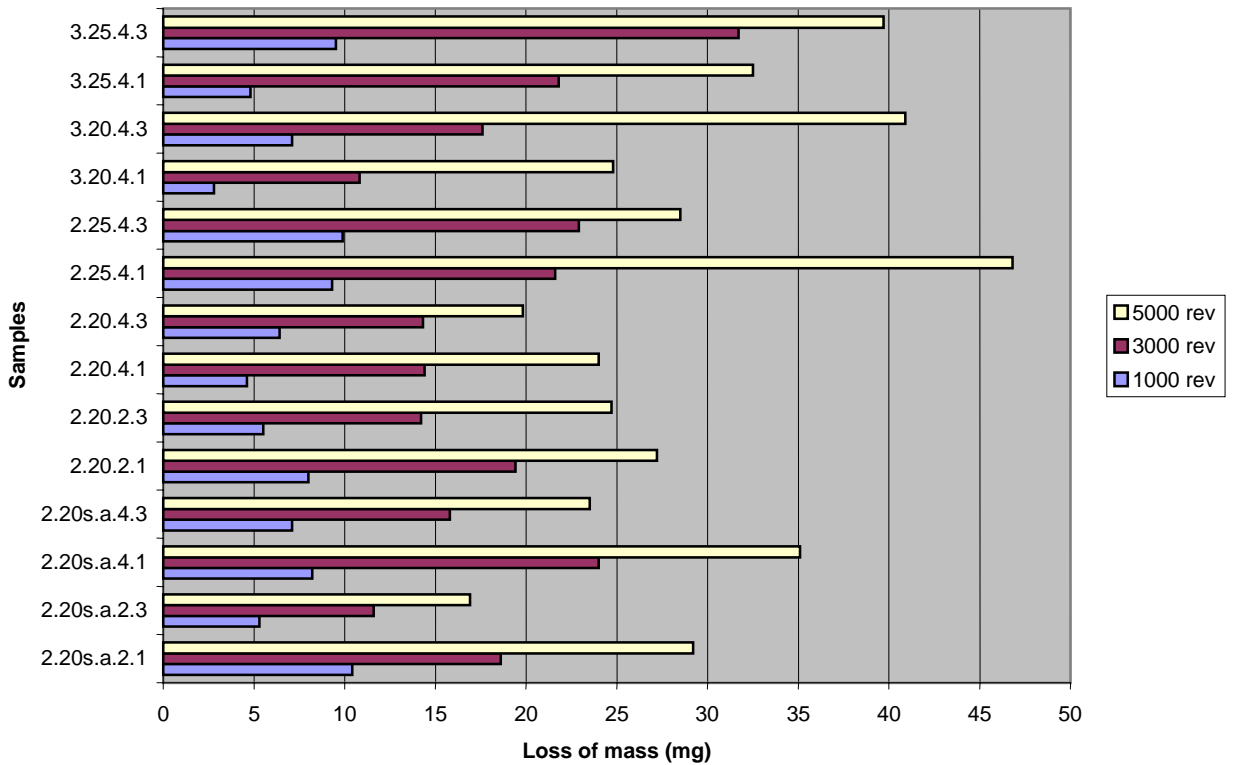
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Fig. 6 – Taber test results expressed as “loss of mass” and “loss of thickness” of the anodic oxidation coating