

EUROPEAN ATOMIC ENERGY COMMUNITY - EURATOM

# MECHANICAL PROPERTIES OF AL-AL<sub>2</sub>O<sub>3</sub> COMPOSITES

Part I : SAP-ISML

by

D.J. BOERMAN, M. GRIN and M. VEAUX

1969



Joint Nuclear Research Center Ispra Establishment - Italy

Metallurgy and Ceramics

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MECHANICAL PROPERTIES OF AL-AL<sub>2</sub>O<sub>3</sub> COMPOSITES PART I: SAP-ISML by D.J. BOERMAN, M. GRIN and M. VEAUX

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Most of the results, described in this report, were realised between 1960 and 1967 in three research centers : JRC-Ispra, ISML, Novara, and Battelle Institut, Frankfurt.

The first chapter describes in short the fabrication process of the semifinished products (bars and tubes) and finished products (canning tubes and pressure tubes). The second chapter deals with the mechanical properties of the SAP-alloys in general. Essentially the chapter consists of :

- talks with results of tension-, compression-, creep- and fatigue tests at various temperatures;
- diagram with the mechanical properties as a function of temperature or percentage of aluminium oxide.

The third chapter describes the experimental conditions and some physical properties of SAP.

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#### ABSTRACT

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#### **KEYWORDS**

MECHANICAL PROPERTIES SAP FABRICATION TESTING TEMPERATURE

### TABLE OF CONTENTS

### Page

а. 1

	INTRODUCTION	1
1.	MANUFACTURING PROCESS	1
1.1 1.1.1 1.1.2	Improvement of the Starting Powders SAP Powders Produced by AIAG SAP Powders Produced by Eckart-We <b>rke</b>	1 1 2
1.2	Cold-compression and Vacuum Treatment	3
1.3 1.3.1 1.3.2 1.3.3 1.3.4 1.3.5	Hot-compression and Extrusion Hot-compression Extrusion Structure of the Extruded Product Definition of the Extrusion Ratio Influence of the Extrusion Ratio on the Mechanical Properties	7 7 9 9
1.4	Drawing	10
1.5 1.5.1 1.5.2	Rolling Hot-rolling Cold-rolling	10 11 11
1 <sup>-</sup> .6	Fabrication of Rods	11
1.7 1.7.1 1.7.2	Fabrication of Cladding Tubes Smooth Tubes Finned Tubes	12 12 13
1.8 1.8.1 1.8.2	Fabrication of Pressure Tubes Extrusion Cold-drawing	13 13 14
2.	STUDY OF THE MATERIAL	14
2.1	Research Laboratories	14
2.2 2.2.1 2.2.2 2.2.3 2.2.4 2.2.5 2.2.6	Presentation of Results Denomination of Material Presentation of Diagrams Symbols Summary of the Main Mechanical Properties Index of Figures Figures	15 15 17 18 20 28
2.2.7	Captions of Figures	30
3.	EXPERIMENTAL CONDITIONS AND COMPLEMENTARY RESULTS	<b>3</b> 7
3.1 3.1.1	Mechanical Properties of Semi-finished Pro- ducts Tensile Tests	37 37

# <u>Page</u>

3.1.1.1 3.1.1.2 3.1.1.3	Euratom and ISML Laboratory Tests AECL Laboratory Tests Risö Laboratory Tests	37 42 42
3.1.2 3.1.2.1 3.1.2.2	Compression Tests (Euratom Results) Experimental Conditions Behaviour of the "Engineering Stress-Strain" Diagrams	43 43 43
3.1.2.3	Results: Cumulative curves for tensile stresses and elongations as a function of the tempera- ture for the four grades of SAP-ISML	44
3.1.3 3.1.3.1 3.1.3.2 3.1.3.3 3.1.3.4	Creep Tests Euratom-ISML Tests Battelle Institute Tests (Frankfurt) AECL Tests Comparison of Results Obtained by Battelle Institute and Euratom	45 45 47 48 48
3.1.4 3.1.4.1 3.1.4.2 3.1.4.3	Relaxation Tests Test Conditions Euratom Results ISML Results	49 49 51 52
3.1.5 3.1.5.1 3.1.5.2	Impact Strength Tests Test Conditions Euratom Results	53 53 53
3.1.6 3.1.6.1 3.1.6.2	Hardness Tests Macrohardness Vickers Microhardness	53 53 54
3.1.7 3.1.7.1 3.1.7.2	Fatigue Tests by Rotating Bending (ISML results) Fatigue Life at 20° and 400°C Notch Effect in Fatigue Tests by Rotating Bending at 20°C	)55 55 56
3.1.8 3.1.8.1 3.1.8.2	Gradual Evolution of Batch Quality (ISML results) Scatter of Results Properties as a Function of Batch Number	57 57 58
3.2	INFLUENCE OF THE MECHANICAL AND THERMAL TREAT- MENTS ON THE EXTRUDED PRODUCT	58
3.2.1 3.2.1.1 3.2.1.2	Influence of Cold-Deformation Tensile Tests Vickers Hardness Tests	59 59 59
3.2.2 3.2.2.1 3.2.2.2	Influence of Heat Treatment After Rolling Tensile Test Vickers Hardness	59 59 60
3.2.3	Influence of the Heat Treatment on SAP 4% at 460°C	60
3.2.4	Influence of Thermal Cycling During Creep Test	60

# <u>Page</u>

3.2.5 3.2.5 1	Influence of Previous Creep Test on Tensile Properties	61 61
3.2.5.2	ISML Results	61
3.3	MECHANICAL PROPERTIES OF FINISHED PRODUCTS	62
3.3.1 3.3.1.1 3.3.1.2 3.3.1.3	Smooth Tubes Tensile Tests (ISML Results) Burst Tests (Euratom Results) Stress-Rupture Tests Under Internal Gas Pressure	62 62 63 64
3.3.2 3.3.2.1 3.3.2.2 3.3.2.3 3.3.2.4	Finned Tubes Tensile Tests Compression Tests Burst Tests Influence of Internal Artificial Defects on the	65 65 66 67
3.3.2.5 3.3.2.6 3.3.2.7	Transverse Mechanical Properties of Finned Tubes Fatigue Tests in Compression Creep Tests in Compression Gradual Evolution of Batch Quality	68 70 71 71
3.3.3	Pressure Tubes	72
3.4	COMPARISON BETWEEN BARS AND TUBES	72
3.4.1 3.4.2	Smooth Tubes Finned Tubes	72 72
3.5	PHYSICAL PROPERTIES	73
3.5.1 3.5.2 3.5.3 3.5.4 3.5.5	Young's Modulus Linear Thermal Expansion Electrical Resistivity Thermal Conductivity Density	73 75 76 78 80
	REFERENCES	81

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#### INTRODUCTION

Under the Orgel Program, Euratom envisaged the possibility of using aluminium alumina composites (Sintered Aluminium Product) for cladding fuel elements (temperature 450°C) and for pressure tubes (temperature 450°C and pressure 20 atm.).

The most important work done on such products with the aim of making them suitable for use in the nuclear field was carried out either under contracts, specially with the ISML (Istituto Sperimentale dei Metalli Leggeri, NOVARA, Italy), and in the laboratories of Euratom's Joint Research Center at Ispra, Italy, especially in the Metallurgy and Ceramics Division.

The research program at ISML was sponsored by Mr. D. GUALANDI (ISML) and Mr. P. JEHENSON (Euratom) (Ref. 1 to 23).

#### 1. MANUFACTURING PROCESS

#### 1.1 Improvement of the Starting Powders (Ref. 22)

The SAP powders used were commercial products which were manufactured by  $AIAG^{\Xi}$  up to 1962, after which they were further developed by the West German firm of Eckart-Werke.

#### 1.1.1 SAP Powders Produced by AIAG

The	SAP	p <b>ow</b> der	s.	of	the	foll	Lov	ving	grade	S
		SAP	96	0	at	oout	4	wt.%	Al <sub>2</sub> 0	3
		SAP	93	0		**	7	11	**	

	<i></i>	•		
SAP	895	"10-11	11	11
SAP	865	"13-14	11	#1

were obtained from 99.5% pure aluminium powders which had undergone fairly lengthy milling at room temperature in a dry oxidizing atmosphere in the presence of stearic acid (Refs. 46-59). Work carried out on several tons of these powders showed that the aluminium oxide content of a specific grade

x Aluminium-Industrie AG (Alusuisse)

- 1 -

may differ between extreme limits of  $\pm 1$  wt.% of Al<sub>2</sub>O<sub>3</sub> from the theoretical percentage for the grade.

The results of two analyses carried out on two batches are given below (wt.%):-

Material	%A1203	% Fe	% Si	% Zn
SAP 930	7.50	0.27	0.12	0.02
SAP 895	10.40	0.27	0.11	0.01

The grain size is generally between 50 and 150 microns. The grains have irregular round shapes and are of spongy aspect (Illustration 1).

The work carried out in 1960-62 on these AIAG commercial powders indicated that it was necessary to obtain better defined powders, especially with regard to the homogeneity of the product (% Al<sub>2</sub>0<sub>3</sub>) and the uniformity of dispersion of the Al<sub>2</sub>0<sub>3</sub> particles in the aluminium matrix.

#### 1.1.2 SAP Powders Produced by Eckart-Werke

The powders were developed by the West German firm in accordance with the basic manufacturing procedures established by AIAG. They are heavy powders the oxide content of which is mainly due to natural oxidation during the milling of the aluminium powder at room temperature. The original aluminium is nuclear aluminium of the order of 99.85% purity.

The aluminium oxide content must be as follows:

SAP 930  $6.5 - 7.5 \text{ wt.} \text{Al}_2 \text{O}_3$ SAP 895  $10 - 11 \text{ wt.} \text{Al}_2 \text{O}_3$ 

The results of two analyses carried out on two batches are given below (wt.%):-

Material	% Al 0 2 3	% Fe	% Si	% Zn	% C
SAP 930	7.25	0.07	0.05	0.01	0.20
SAP 895	10.35	0.08	0.06	0.01	0.25

#### 1.2 Cold-compression and Vacuum Treatment (Ref. 22, Ill. 2)

Commercial products exposed to high temperatures (500-600<sup>o</sup>C) for several hours showed surface blisters and internal microcracks, and consequently insufficient structural stability for nuclear uses (Ref. 36). This serious drawback was due to the high residual gas content (mainly hydrogen) of the finished products. While the lowering of the hydrogen content is of fundamental importance, a study has been in progress since 1960 aimed at eliminating this gas by vacuum treatment. The treatment selected, described in patent No. 13.532, registered in Italy on 30 May 1961, has the following main features:

- cold-compression of the powders at about 20 kg/mm<sup>2</sup> before heating in the vacuum furnace;
- treatment in the 590-620°C range for a period of 20-24 hr;
- vacuum between  $10^{-4}$  and  $10^{-5}$  mm Hg.

The extruded products contain only a small amount of residual gas, the quantity of which seems to be a function of the oxide content.

Mater	ial	Gas content
SAP ISML	4% Al	2 <sup>0</sup> 3 1-3
SAP ISML	7% '	2-5
SAP ISML	10% '	3-6
SAP ISML	14% '	4-8



Illustration 1 Structural aspect of Al-Al<sub>2</sub>0<sub>3</sub> powders grade 960 Etching HF 0.5% Magnification 500 x



extrusion direction



Illustration 3

Longitudinal microstructure of bars of SAP ISML 7% Magnification 500 x 2 Etching HF 0.5% 1.3 Hot-compression and Extrusion (Ref. 23, Ill. 2)

#### 1.3.1 Hot-compression

After the vacuum treatment the billet, wrapped in an aluminium foil, is immediately inserted in the container of the press and subjected to a specific pressure of 50 kg/mm<sup>2</sup> for 30 min.

The process is carried out with a 250 t press for billets 80 mm in diameter and with one of 500 t for billets 110 mm in diameter. At the end of the process, the density of the product is of the order of 2.7  $g/cm^3$ . The crust is then removed and the billet pickled.

#### 1.3.2 Extrusion

The compacted billets (diameter 110 or 80 mm) are preheated at a temperature of between 540 and  $590^{\circ}$ C for 3 hr. The temperature of the extrusion container is about  $330^{\circ}$ C. The process is carried out on one of the three presses, the characteristics of which are given below:-

Max. force tons	Diameter of starting billet (mm)	Utilization
250	80	smooth tubes rods
500	80 110	finned tubes rods
2700	250	pressure tube

The extrusion velocity varies between 8 m/sec (pressure tube) and 16 to 20 m/sec (rods and finned tubes). In order to obtain rods and tubes, a double extrusion is usually carried out.

The extruded product constitutes the starting material for the rolling and cold drawing processes, which followed in some cases by heat treatment, lead to the finished products.



<u>Illustration 4</u> Electron micrograph of a bar of SAP-ISML 7% Al<sub>2</sub>0<sub>3</sub> Electron microscope magnification x 8,000

Total magnification x 20,000

#### 1.3.3 Structure of the Extruded Product

The extruded product seems to have a very fine dispersion of  $Al_2O_3$  particles inside the aluminium matrix. The dimension of the particles is between 0.02 and 0.1 micron and the interparticle distance is of the order of 0.1 to 0.2 micron (see Il-lustrations 3 and 4).

#### 1.3.4 Definition of the Extrusion Ratio

Any time extruded products (rods, thick-walled tubes, finned tubes) are handled, the strictest definition we can give is the following:-

$$E^* = \frac{A_1}{A_2}$$

$$A_1^* \text{ section of the container}$$

$$A_2^* \text{ section of the die}$$

However, in current practice another relation which gives quite reliable results is used:

73	
$E = S_2$ $S_2$ : section of the extra section of t	ruded

It is the latter relation which will be taken into consideration in our study.

#### 1.3.5 <u>Influence of the Extrusion Ratio on the Mechanical</u> <u>Properties</u>

It is interesting to note that the value E is not sufficient to characterize the state of deformation of the material. For example, a finned tube or a round rod extruded at the same extrusion ratio will not give an equivalent strain hardening and consequently the mechanical properties will not be comparable. For instance, the final extrusion ratio for rods  $\emptyset$  9.5 and for finned tubes are approximately the same (comprised between 70 and 80). However, the longitudinal strength of finned tubes is 18% higher (see Section 3.4.2 and Histograms Fig. 117).

In order to examine this influence in the simple case of rods, we performed tensile tests at 20 and 450°C on rods of SAP ISML 7% and 10% of various extrusion ratios respectively prepared from the same batch of powder. The characteristics of the material are listed in the table below:-

%Al <sub>2</sub> 0 <sub>3</sub>	Bar Ø	Extrusion	Batch No.	Batch No.
	(mm)	ratio	(extrusion)	(powder)
7%	9.5	76	3,504/3	M 133
	16	27	3,448/1	M 129
	21	14	3,447/1&2	"
	23	13	3,504/1&2	M 133
10%	9.5 16 21 23	76 27 14 13	3,240/1 3,239-3,240/2 3,237-3,238 3,234-3,235-3,236	<b>№</b> 4041

For the 9.5 mm  $\emptyset$  bar, the test specimen was as represented on Fig. 3, but for the others, the cross section was greater in order to maintain constant the ratio

# $R = \frac{\text{specimen cross section}}{\text{bar cross section}} = 0.18$

However, for all specimens, the ratio <u>length</u> = 7.5 was maindiameter tained constant (see Section 3.1.1.1.1 and Fig. 1.2.3).

The results obtained show that the extrusion ratio influences only the values of elongation at breaking point ( $e_{pb}$ ).

#### 1.4 <u>Drawing</u> (Ref. 23)

This procedure has mainly been used for the fabrication of smooth tubes and pressure tubes. The deformation ratio as a percentage, obtained either by drawing or by rolling without intermediate annealing, is given by the following relation:

 $H = \frac{S - s}{s} \times 100$  H : ratio of cold-deformation in % S : initial section of the material s : final section of the material

1.5 <u>Rolling</u> (Ref. 23)

The starting material consists of plates of  $50 \ge 12 \text{ mm}$  or  $30 \ge 2.5 \text{ mm}$ . These are either directly cold-rolled or partly hot-rolled ( $40 \le H \le 70\%$ ) and then finished at room temperature.

#### 1.5.1 Hot-rolling

The extruded rough shapes are first preheated to  $530^{\circ}$ C for 24 hr and then rolled transverse to the extrusion direction until a deformation ratio of 50% is obtained, and then parallel to the extrusion direction until a ratio of 70% is reached.

#### 1.5.2 <u>Cold-rolling</u>

This is carried out either directly on the extruded material or on materials that have already undergone hot-rolling deformation. The table below gives the deformation ratios obtained by the two methods:

Deformation ratio values (%)								
%A1203	hot-rolling	extrusion						
4	73	_						
7	37	<b>3</b> 2 ·						
10	<b>19</b>	20						
14	10	8						

Before cold-rolling, an annealing treatment of 2 hr at  $530^{\circ}$ C is necessary. In the case of SAP ISML 4 and 7% deformation ratios of 99% are obtained.

#### 1.6 <u>Fabrication of Rods</u> (Ref. 23)

Up to the end of 1965 (batch No. 2500) the starting billet (hot-compacted) had a diameter of 78.5 mm. The results of the mechanical tests on rods presented in this document concern only the extruded materials made from billets 78.5 mm in diameter.

#### Characteristics of the first extrusion

diameter of container: 80 mm dimension of starting billet: 78.5 x 140 mm diameter of billet obtained: 58 mm extrusion ratio: E = 1.9extrusion rate: 1.3 m/min temperature: 568°C Afterwards the second extrusion is carried out directly on the final diameter of the rod with the same temperature conditions. The extrusion ratios relating to the various diameters of rods extruded from compacts of 0.D. 80 mm are given in the following table.

1st Ex	trusion	2nd Ex	trusion	Final		
Diameter (mm)	Extrusion ratio	Diameter (mm)	Extrusion ratio	ratio		
		23	6.8	13		
		_ 21	8.2	14		
58	1.9	16	, 14	26.6		
		12	25	47.5		
		9.5	40	76		

#### 1.7 Fabrication of Cladding Tubes (Ref. 23)

The smooth tubes are fabricated by extrusion followed by colddrawing, the finned tubes by extrusion only.

#### 1.7.1 Smooth Tubes

Since the mechanical tests were only carried out on tubes  $13.1 \times 14.7$ mm in diameter we can limit ourselves to indicating the fabrication conditions of smooth tubes of these dimensions only.

### 1.7.1.1 Extrusion

The starting material is a cylindrical hollow billet through which a lubricated, slightly conical mandrel is passed which is of the same diameter as the internal diameter of the tube after extrusion (extrusion by free floating mandrel).

In the case considered here, the extruded tube has diameters of 16 and 14 mm (E = 59). The extrusion temperature is  $571^{\circ}$ C and the velocity is between 16 and 20 m/min.

#### 1.7.1.2 Cold-drawing

The cold-drawing characteristics for the tubes concerned are given below:-

	diameter of die (mm)	diameter of mandrel (mm)	wall thickness of tube (mm)
1st step 2nd step	15.0 14.7	13.4 13.1	0.8

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an an g

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1.7.2 Finned tubes (Ref. 23)

The extrusion is carried out in two steps on a 500 t press at a temperature of  $575^{\circ}C$  starting from a hot-compacted billet 110 mm in diameter. The first extrusion gives a billet either 80 mm in (E = 2) diameter of 60 mm (E = 4). The extrusion rate is 1.3 m/min. Afterwards a central hole with a diameter equal to the final internal diameter of the hole is drilled. A second extrusion at rates between 16 and 20 m/min results in the desired profile. The extrusion ratios are between 17 and 20. The method is the same as that applied for extruding smooth tubes (free floating mandrel). The fins may be straight or helicoidal.

### 1.8 Fabrication of Pressure Tubes

These are tubes of large diameter (about 100 mm). As in the case of smooth tubes, they are first extruded and then cold-drawn.

#### 1.8.1 <u>Extrusion</u>

The process is carried out at a temperature of  $570^{\circ}$ C on a 2700 t press followed by annealing for 2 hr at  $540^{\circ}$ C. The extrusion rate is of the order of 8 m/min. The dimensional characteristics are listed in the table below:

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#### dimension of the extruded tube

	before	extrusion	after extrusion				
	ID mm	OD mm	ID mm	OD mm			
1st extrusion 2nd extrusion	98 97	250 198	97 96	198 102			

At the end of the process the extruded tube is annealed for 2 hr at  $540^{\circ}C$ .

#### 1.8.2 Cold-drawing

The cold-drawing is carried out in two steps; after each step annealing for 2 hr at  $540^{\circ}$ C is necessary. The dimensional characteristics are given below:-

dimension of the drawn tube

	before c	old-drawing	after c	old-drawing
	ID mm	OD mm	ID mm	OD mm
1st step 2nd step	96 94.05	102 100.4	94.05 92.2	100.4 98.4

#### 2. STUDY OF THE MATERIAL

#### 2.1 <u>Research Laboratories</u>

Euratom has undertaken studies on the improvement of this material in the laboratories of the Ispra Joint Research Center and has concluded contracts with several companies.

With the Italian firm of Montecatini, it has drawn up two contracts: a research contract to be carried out in the laboratories of the Istituto Sperimentale dei Metalli Leggeri (ISML), Novara, and a contract for the production of bars, cladding tubes and pressure tubes on a semi-industrial scale.

A research contract concerning creep tests has also been concluded with the Battelle Institute, Frankfurt, Germany.

Some other results, reported here, were obtained by the Danish Atomic Energy Commission at its Risö research establishment. Other tests were conducted by the Civilian Atomic Power Department of the Canadian General Electric Company Ltd. (Peterborough, Ontario) and reprinted in the AECL publications (Atomic Energy of Canada Ltd., Chalk River, Ontario).

#### 2.2 Presentation of Results

The main results will be presented in the form of diagrams whenever possible; the experimental conditions, the comments and some complementary results in tabular form will be found with all the references in an appendix at the end of the report.

#### 2.2.1 Denomination of Material

The material is characterized by its percentage number (4, 7, 10 or 14% principally). This refers to the nominal percentage of  $Al_2O_3$  by weight in the aluminium matrix. Often the real weight percentage differs by at least  $\pm 1 \text{ wt.\% of } Al_2O_3$ . The results refer only to SAP produced in standard conditions (see Section 1). In addition, all the results concern SAP manufactured by ISML, which is named SAP ISML in order to distinguish it from, for example: SAP AIAG

FRITTOXAL (TLH) APM alloys, etc.

The material is also given a batch number at the beginning of the fabrication process. The batch numbers are allocated chronologically, so this number indicates the fabrication period. The principal fabrication characteristics for a given batch number are collected in tables published in the quarterly progress reports of ISML, Novara.

Table 1 gives a list of the batch numbers for the specific quarterly progress reports and for the various types of powders used.

_		
mo	bl o	- 1
⊥a	JULE	

Fabrication Data of the Various Batches of SAP

No. of Batch	Date of fabrication	Ref.	Type of powder employe				
1 82 128 173 251 347 441 555 734 851 926 1137 1241 1344 1636 1831 1939 2209 2353 2483 2680 3172 3440	1 - 5 - 1960 $30 - 7 - 1960$ $31 - 10 - 1960$ $15 - 2 - 1961$ $15 - 5 - 1961$ $31 - 7 - 1961$ $30 - 11 - 1961$ $28 - 2 - 1962$ $31 - 5 - 1962$ $30 - 9 - 1962$ $31 - 12 - 1962$ $30 - 4 - 1963$ $31 - 12 - 1963$ $30 - 6 - 1964$ $31 - 10 - 1964$ $31 - 10 - 1964$ $31 - 12 - 1965$ $31 - 8 - 1965$ $31 - 8 - 1965$ $31 - 12 - 1965$ $30 - 4 - 1965$ $31 - 12 - 1965$ $30 - 4 - 1966$ $31 - 8 - 1966$ $31 - 8 - 1966$ $31 - 12 - 1966$	<ul> <li>1</li> <li>2</li> <li>3</li> <li>4</li> <li>5</li> <li>6</li> <li>7</li> <li>8</li> <li>9</li> <li>10</li> <li>11</li> <li>12</li> <li>13</li> <li>14</li> <li>15</li> <li>16</li> <li>17</li> <li>18</li> <li>19</li> <li>20</li> <li>21</li> <li>21B</li> </ul>	4 % ↓ A⊥AG	7% J Ecka and Ecka	10% J IAG rt-Werke AIAG rt-Werke	14%	
3740 3827	30 - 4 - 1967 31 - 8 - 1967	210	4%	↑ 7%	10%	14%	

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#### 2.2.2 Presentation of Diagrams

Normally metric units were used, but whenever the references employed British units, both are reported. As far as possible standard scales were adopted for the different graphs in order to facilitate comparison. The dimensions of specimens often influence the results, so they were given. For every cumulative diagram, reference is made to the figure numbers in the original individual diagrams and their respective document reference numbers.

The general scheme of presentation of the results will be the following:

- a table and a figure (Table 2 and Illustration 5) show all the symbols and terms used in English, French and German to characterize the mechanical properties in tensile and compression testing.

- seven tables summarize the main mechanical properties of the four grades of SAP for bars, smooth tubes and finned tubes (Tables 3 to 9).

- an index of all the measured mechanical and physical properties of SAP with reference to the corresponding figures (Tables 10 and 11).

- the figures gathered according to the type of test: Semi-finished products

. Mechanical properties

Tensile tests Compression tests Creep tests Relaxation tests Impact strength tests Hardness tests Fatigue tests Evolution of rod production

. Physical properties Finished products

> Various mechanical tests like tensile tests, compression tests, burst tests and fatigue burst tests.

# SYMBOLS



# <u>Table 2</u>

Symbol	Definition	Unit	English	French	German
P <sub>pr</sub>		kg	Proportional elastic load	charge à la lìmite de proportionnalité	Kraft an der Proportionali- tätsgrenze
P0.2		kg	0.2 offset yield load	charge à la limite élastique à 0,2%	Kraft an der 0,2 Streck- grenze
Pu		kg	upper load	charge maximum	Maximum-Kraft
Ao		mm <sup>2</sup>	original gauge cross- section	section originale	originaler Querschnitt
L pu		mm	plastic uniform elongation	allongement plastique réparti	plastische Gleichmass- verlängerung
L <sub>pb</sub>		mm	plastic elongation at breaking point	allongement plastique à la rupture	plastische Verlängerung beim Bruch
Lo		mm	original gauge length	base de mesure	originale Messlänge
Spr	P <sub>pr</sub> /A <sub>o</sub>	kg/mm <sup>2</sup>	engineering proportional elastic limit	contrainte à la limite de proportionnalité	technologische Proportiona- litätsgrenze
<sup>S</sup> 0.2	P0.2/A0	kg/mm <sup>2</sup>	engineering 0.2 offset yield stress	limite élastique à 0,2%	technologische 0,2 Streck- grenze
S <sub>u</sub>	P <sub>u</sub> / A <sub>o</sub>	kg/mm <sup>2</sup>	engineering maximum tensile stress	contrainte maximum	technologische Zugfestig- keit
e <sub>pu</sub>	$\frac{\Delta L_{pu}}{L_{o}} \ge 100$	%	engineering plastic uniform strain	allongement plastique (unitaire) réparti	technologische plastische Gleichmassdehnung
e <sub>pb</sub>	$\frac{\Delta L_{pb}}{L_{o}} \times 100$	%	engineering plastic strain at breaking point	allongement plastique unitaire à la rupture	technologische plastische Bruchdehnung
Е	<u>5</u> e	kg/mm <sup>2</sup>	Young's Modulus of elasticity	module élastique (module d'Young)	Elastizitätsmodul



# SUMMARY OF THE MAIN MECHANICAL PROPERTIES



# MAIN MECHANICAL PROPERTIES

Type of Material	Table
bars SAP 4%	3
bars SAP 7%	4
bars SAP 10%	5
bars SAP 14%	6
smooth tubes SAP 4%	7
smooth tubes SAP 7%	8
finned tubes SAP 7%	9

<u>Table 3</u>

### Main mechanical properties of SAP ISML 4%

Type of test	Room tem	perature p	proper	ties.		High temperat					ture properties				
Tensile	S <sub>u</sub> (kg/mm <sup>2</sup> )	S <sub>0.2</sub> (kg/mm <sup>2</sup> )	e <sub>pu</sub> (%)	epb (%)	Test tempera- ture <sup>o</sup> C	S <sub>u</sub> (kg/mm <sup>2</sup> )	S <sub>0.2</sub> (kg/mm <sup>2</sup> )	e <sub>pu</sub> (%)	epb (%)	Test tempera- ture °C	S <sub>u</sub> (kg/mm <sup>2</sup> )	<sup>S</sup> 0.2 (kg/mm <sup>2</sup> )	e <sub>pu</sub> (%)	epb (%)	11-12 (Eur) 20-22-25-28
test (1)	20.0 24.5	13.2 18	9.6 12.2	19.8 25.8	400	5.9 6.5	4.5 6.2	0.6 0.7	2.5 11.0	450	5.0 6.1	4.0 6.0	0.6	4.3 9.6	(ISML) 32-33 (AECL) 34 (Risö)
Compres- sion tests	34.8 32.8	21.2 19.6	16.0 15.3		400	7.8	6.2	9.0		450	6.7	6.0	6.7		35-36-37
Creep tests (Maximum ad- missible					Max. (kg	Max. stress Elongation at (kg/mm <sup>2</sup> ) rupture (%)		Max. stress (kg/mm <sup>2</sup> )		Elongation		40			
stress for 10,000 hrs life).					400 426	4	•3 •1			460	3.2	± 0,.1	0.5	± 8:2	55 <b>-</b> 56 - 60 ⊷
Fatigue limit in	Maximu	m stress	(kg/mm	<sup>2</sup> )		Maximum stress (kg/mm <sup>2</sup> )			'mm <sup>2</sup> )					75	
rotating bending		8.5		400 4.8											
Young's Modulus (kg/mm <sup>2</sup> )		7,200			400	400 4,400 ± 200		450 4,100 ± 100				93			

(1) 6 to 15 specimens for each temperature
### Main Mechanical Properties of SAP ISML 7%

<u>Table 4</u>

.

Type of test	Room temy	perature j	proper	ties			High	temper	ature p	roperties					References of Figures
Tensile	S <sub>u</sub> (kg/mm <sup>2</sup> )	S <sub>0.2</sub> (kg/mm <sup>2</sup> )	e <sub>pu</sub> (%)	e <sub>pb</sub> (%)	Test tempera- ture C	S <sub>u</sub> (kg/mm <sup>2</sup> )	<sup>S</sup> 0.2 (kg/mm <sup>2</sup> )	e <sub>pu</sub> (%)	epb (%)	Test tempera- ture C,	S <sub>u</sub> (kg/mm <sup>2</sup> )	<sup>S</sup> 0.2 (kg/mm <sup>2</sup> )	e <sub>pu</sub> (%)	e <sub>pb</sub> (%)	13-14 (EUR) 20-22-25-28
tests <sup>(1)</sup>	20.0 25.5	14.3 20.4	7.9 12.7	15.0 23.2	400	8.3 9.0	7.8 8.1	0.7 0.8	1.8 6. <u>3</u>	450	7.5 7.8	7.0 7.6	0.4 0.6	3.5 6.4	(ISML) 34 (Risö)
Compres- sion tests	40.3 40.0	22.7 22.0	20.8 18.7		400	9.4	8.3	5.7		450	8.2	7.2	5.3		35-36-37
Creep tests (Maximum ad-					Max. stress Elongati (kg/mm <sup>2</sup> ) rupture		tion at re (%)	Max. stress (kg/mm <sup>2</sup> )		stress /mm <sup>2</sup> )	Elon at r <b>u</b>	gation upture (%)	41-42 <b>43-</b> 44		
missible stress for					400	400 5.8				460	4.5 ± 0.2		0.6	+ 0.8 - 0.3	55-57-58
10,000 hrs. life).	¶ 				420	5	± 0.3								60
Fatigue limit in	Maximur	n stress	(kg/mm	<sup>2</sup> )		Maxim	um stress	(kg/m	1						
bending		7.5			400	0 6									76
Young's Modulug (kg/mm <sup>2</sup> )		7,200			400	400 5,000 ± 50 0			450	4,700 ± 50				93	

-

(1) 5 to 10 specimens for each temperature

- 22 -

### Main Mechanical Properties of SAP ISML 10%

<u>Table 5</u>

Type of test	Room tem	perature p	roper	ties		_	High te	mp <b>er</b> a	ture p	propertie	s				References
Tensile	S <sub>u</sub> (kg/mm <sup>2</sup> )	<sup>S</sup> 0.2 (kg/mm <sup>2</sup> )	e <sub>pu</sub> (%)	epb (%)	Test tempera- ture C	S <sub>u</sub> (kg/mm <sup>2</sup> )	S <sub>0.2</sub> (kg/mm <sup>2</sup> )	e <sub>pu</sub> (%)	e <sub>pb</sub> (%)	Test tempera- ture C	Su (kg/mm <sup>2</sup> )	S <sub>0.2</sub> (kg/mm <sup>2</sup> )	e pu (%)	e pb (%)	15-16 (Eur)
tests <sup>(1)</sup>	29.8	21.5	6.9	12.0	400	11.0	9.4	0.3	1.0	450	9•5	8.0	0.3	2.6	20-22-25-28 (ISML)
	30.8	24.8	8.2	18.0		12.5	12.0	0.4	3.8		10.0	9.9	0.6	5.2	34 (Risö)
Compres- sion tests	47.6 46.4	28.6 26.7	21.3 21.0		400	11.6	10.3	7•7		450	10.1	8.9	6.0		35-36-37
Creep tests (Maximum ad-						Max. (kg	stress /mm <sup>2</sup> )	Elonga ruptur	tion at e (%)		Max. : (kg/	stress (mm <sup>2</sup> )	Elon/ at rup	gation ture(%)	
missible stress for					400	6	.8			460	5	•9 ;	,		45
10,000 hrs life).		<u></u>	<u></u>		426	6	•2								60
Fatigue limit in	Maximur	n stress (	kg/mm	<sup>2</sup> )		Maximum	stress (	kg/mm	<sup>2</sup> )						78
rotating bending	9.5				400	5.8								[]	
Young's Modulus (kg/mm <sup>2</sup> )		7,500			400		5,150 ±	150		450	4,850			93	

(1) 6 to 11 specimens for each temperature

### Main Mechanical Properties of SAP ISML 14%

#### <u>Table 6</u>

.

Type of test	Room tem;	perature	proper	rties			High	tempe	ratur	e propert	ies			•	References of Figures
Tensile	S <sub>u</sub> (kg/mm <sup>2</sup> )	\$0.2 (kg/mm <sup>2</sup> )	e pu (%)	epb (%)	Test tempera- ture C	S <sub>u</sub> (kg/mm <sup>2</sup> )	S <sub>0.2</sub> (kg/mm <sup>2</sup> )	e <sub>pu</sub> (%)	epb (%)	Test tempera-	S <sub>u</sub> (kg/mm <sup>2</sup> )	<sup>S</sup> 0.2 (kg/nm <sup>2</sup> )	e <sub>pu</sub> (%)	epb (⅔)	17-18 (Eur) 20-22-25-28
Tests <sup>(1)</sup>	36.0 37.7	26.0 30.7	6.6 7.0	8.0 12.0	400	14.0 14.5	12.0 14.3	0.3 0.5	0.8 2.3	450	11.8 13.0	10.5 12.6	0.3 0.4	1.0 2.6	(ISML) 34 (Risö)
Compres- sion tests	57.2 47.6	34.7 33.6	21.3 19.0		400	14.2	12.3	5.0		450	12.3	11.2	4.3		35-36-37
Creep tests (Maximum ad-						Max.	stress g/mm <sup>2</sup> )	Elonga rupture	tion at (%)		Maximu (k	m stress g/mm <sup>2</sup> )	Elon at ruj	gation pture (%)	
stress for 10,000 hrs. life).					400		7.5		•	450		7.1			46
Fatigue limit in	Maximu	n stress	(kg/m	1 <sup>2</sup> )		Maxim	um stress	(kg/m	m <sup>2</sup> )						
rotating bending		12			400		7								78
Young's Modulus (kg/mm <sup>2</sup> )	7,700		400	5,300 ± 50		450	5,100 ± 50			93					

(1) 4 to 6 specimens for each temperature.

### Main Mechanical Properties of Smooth Tubes in SAP ISML 4%

### <u>Table 7</u>

Type of test	Mechanical and thermal treat- ment	Room temperature properties			High te	Reference figures			
Tensile	Extruded then	S <sub>u</sub> (kg/mm <sup>2</sup> )	S <sub>0.2</sub> (kg/mm <sup>2</sup> )	epb (%)	Test tempera- ture °C	S <sub>u</sub> (kg/mm <sup>2</sup> )	<sup>S</sup> 0.2 (kg/mm <sup>2</sup> )	e <sub>pb</sub> (%)	
test	ameared	22,5		14	450	6.0	4.5	5 <b>±1</b>	105
	Extruded, Cold-drawn, then annealed	25	22.3	8	450	5.0	4.0	5 <b>±</b> 1	106 107
Creep under internal gas	Annealed				400	Maximum for a li	stress (k lfe of 2,0 3.1	$(g/mm^2)$	108 109
pressure				450	1.9	± (	).6		

### <u>Table 8</u>

## Main mechanical properties of

smooth tubes in SAP ISML 7%

Type of test	Mechanical and thermal treatment of the material	Room temperature properties			High temperature properties				References of figures
Tensile	Extruded then	S <sub>u</sub> (kg/mm <sup>2</sup> )	S <sub>0.2</sub> (kg/mm <sup>2</sup> )	epb (%)	Test tempera- ture C	$ \begin{bmatrix} S_u \\ (kg/mm^2) \end{bmatrix} \begin{bmatrix} S_{0.2} \\ (kg/mm^2) \end{bmatrix} \begin{bmatrix} e_{pk} \\ (\%) \end{bmatrix} $		<sup>e</sup> pb (%)	
Test	annealed	27.3	14.5	10	450	8.7	6.5	3 <b>±0.</b> 8	105
	Extruded, Cold-drawn then annealed	30	2 <b>2</b>	5	450	6.8	4.5	3 <b>±</b> 0.8	106
Creep under						Maximum life of	stress f 1,000 hr	or a s.	
internal gas	Annealed				400		4.1		110
pressure					450		3.5		
	Cold-drawn				400		4•4	_	111
	Cold-drawn then annealcd				400		3.2	<u>.</u> .,	]

## Main high temperature mechanical properties

of finned tubes in SAP ISML 73

Table 9

Type of test	Test tempera- ture C	Rupture stress (kg/mm <sup>2</sup> ) Elongat:			Elongati	on at rup	ture (%)	References of Sections and Figures
		Min. value	Mean value	Max. value	Min. value	Mean value	Max. value	
Tensile tests	450	6.8	9	10.8	1	3.2	5	Fig. 112
Compression tests	450	6.9	7	7.4	0.16 <sup>(1)</sup>	0.26 <sup>(1)</sup>	0.39 <sup>(1)</sup>	Sect. 3.3.2
Burst test (tangential stress)	460	5.1	5.6	6.0			į.	Sect. 3.3.2
Burst test on tubes with internal artificial de- fects (15% wall thickness	460	5.0	5.3	5.55				Sect. 3.3.2 Fig. 118
Fatigue tests with in- ternal gas pressure (with and without in- ternal defects)	460	4.1(2)	4.3(2)	4.45 <sup>(2)</sup>				Sect. 3.3.2 Fig. 119
Creep tests in compres- sion	460		<b>€</b> 4 <sup>(3)</sup>					Sect. 3.3.2

(1) In this case, the elongation corresponds to  $e_{pu}$ .

(2) For a life to rupture of 5,000 cycles.

(3) For a life to rupture of about 5,000 hours.

# INDEX OF FIGURES



Table 10 Index of Figures Mechanical Properties of Semi-finished Products

Type of test	Figures	as a function of t for each grade of f	temp <b>er</b> ature SAP		Figures as a oxide	function of content
	SAP 4%	SAP 7%	SAP 10%	SAP 14%	Room temp.	High temp.
Mensile	11-12-29	13-14	15-16	17-18	10-28-34	10-28-34
TOUBLE	<u>19-20-21-22</u> 23-24-25	<u>19-20-21</u> 22- <u>23</u> - <u>24</u> - <u>25</u>	<u>19-20-21</u> 22-23-24-25	<u>19-20-21-22</u> 23-24-25	<u>26-27</u>	<u>26-27</u>
Compression	<u>35-36-37</u>	<u>35-36-37</u>	<u>35-36-37</u>	<u> 35-36-37</u>		
	40-52-55-56	41-42-43-44	45	46		
Creep	<u>48-49-50-51-60</u> <u>61</u>	55-57-58 47-48-49-50 51-60-61	<u>48-49-50-51-60</u>	<u>48-49-50-51</u>		
Relaxation		64-65a-66 <u>65</u>	<u>65</u>			
Impact	<u>68</u>	<u>68</u>				
Hardness	<u>69a</u> -72-70a- <u>71-73</u>	<u>69a-70a-70b-71-73</u>	<u>69a-70a-71-73</u>	<u>70a-70b-71-73</u>	69-70	
Fatigue	75-82- <u>79</u>	76-83- <u>79</u>	77-84- <u>79</u>	78-85- <u>79</u>		
Evolution of rod production	<u>86-87</u>	88-89-90-91 <u>86-87</u>	<u>86-87</u>	<u>86-87</u>		

NB: Underlined numbers refer to cumulative figures.

### Physical properties of semi-finished products

Property or Test	Figures	as a functi each gra	on of temper de of SAP	ature for	Figures as a function of oxide content		
	SAP 4%	SAP 7%	SAP 10%	SAP 14%	Room temp.	High temp.	
Young's Modulus	<u>93</u>	<u>93</u>	<u>93</u>	<u>93</u>	92		
Coefficient of thermal expansion					<u>94</u>	<u>94</u>	
Electrical resistivity	<u>96-97</u>	<u>96-97</u>	<u>96-97</u>	<u>96-97</u>	95	98	
Thermal conductivity	<u>99-100-101</u>	<u>99-100-101</u>	<u>99-100-101</u>	<u>99-100-101</u>		102 - 103	
Density					104 - 104A,		
		-	Ja				

### Mechanical properties of finished products

Tensile tests on smooth tubes		105-106-107	105-106-107
Creep tests with internal gas pressure on smooth tubes	108-109-115 110-111-116		
Tensile tests on finned tubes	112-113-114 117		

NB: Underlined numbers refer to cumulative figures.

# FIGURES



## - 30 -

## 2.2.7 <u>Captions of Figures</u> <u>SEMI-FINISHED PRODUCTS</u>

## Tensile Tests

Fig.	
1 and 2	Influence of the gauge length on the engi-
	neering plastic elongation at breaking point
	during tensile test.
3	Euratom tensile specimen (TR1)
4.	ISML tensile specimen
5	Influence of the extrusion ratio on the
	elongation e pb
6	Influence of test duration on the mechanical
	properties
7,8,9	Influence of the crosshead speed on the me-
9A,9B,9C	chanical properties
10	The elastic limit of SAP ISML at $20^{\circ}$ and $400^{\circ}$ C
	obtained through different methods
11	Tensile tests on SAP 4% Stress $S = f(T)$
12	Tensile tests on SAP 4% Strain $e = f(T)$
13	Tensile tests on SAP 7% Stress $S = f(T)$
14	Tensile tests on SAP 7% Strain $e = f(T)$
15	Tensile tests on SAP 10% Stress $S = f(T)$
16	Tensile tests on SAP 10% Strain $e = f(T)$
1.7	Tensile tests on SAP 14% Stress $S = f(T)$
18	Tensile tests on SAP 14% Strain $e = f(T)$
19	Cumulative Euratom results Stress $S_u = f(T)$
20	$Cumulative ISML results Stress S_{u} = f(T)$
21	Cumulative Euratom results Stress $S_{0.2} = f(T)$
22	Cumulative ISML results Stress $S_{0.2} = f(T)$
23	Cumulative Euratom results Strain $e_{pu} = f(T)$
25	Cumulative Euratom results Strain $e_{pb} = f(T)$
25	Cumulative ISML results Strain $e_{pb} = f(T)$
20	Cumulative results Stress S & S $0.2 = f(\% 1 0)$
<i>2</i>	cumulative results strain $e_{pb} \approx e_{pu} = f(%A1_2_{03})$

Fig.	
28	Tensile properties as a function of the oxide
	content at 20 and 400 <sup>0</sup> C (ISML)
29	Results for SAP 4% after different transformations
	Stress $S = f(T)$
30	Results for SAP 4% after different transformations
	Strain $e = f(T)$
31	AECL tensile specimen

32 AECL results on SAP 4% Stress S = f(T)33 AECL results on SAP 4% Strain e = f(T)34 Curves as a function of percentage of Al<sub>2</sub>O<sub>3</sub> at 20 and 400°C (Risö data)

### Compression Tests

35	Cumulative	results	Stress	s <sub>u</sub> ' =	f(T)
36	Cumulative	results	Stress	s <sub>0'2</sub>	= <b>f(</b> ])
37	Cumulative	results	Strain	e pu	= f(T)

### Creep Tests

38	Euratom creep specimen
39	ISML creep specimen
40	Stress-rupture curves of SAP 4% at 400 and $460^{\circ}$ C
41	Stress-rupture curves of SAP $7\%$ (old batches) at
	400 and 460 <sup>°</sup> C
42	Stress-rupture curves of SAP 7% (batch 1722) at 420°C
43	Stress-rupture curves of SAP $7\%$ (old and recent
	batches) at 460°C
44 .	Scatter of strain-values of SAP $7\%$ at $460^{\circ}$ C
45	Stress-rupture curves of SAP 10% at 400 and 460°C
46	Stress-rupture curves of SAP 14% at 400 and 460°C
47	Cumulative stress-rupture curve of SAP 7% at dif-
	ferent temperatures.

Fig.	
48	Cumulative stress-rupture curves of different SAP grades at 400°C (ISML results)
49	Cumulative stress-rupture curves of different SAP grades at 460°C (Euratom results)
50	Stress to produce 0.1% creep elongation at 400°C (ISML results)
51	Creep extension after 1,000 hours at 400°C (ISML results)
52	Stress-rupture curves of SAP 4% at 460 <sup>°C</sup> after heat treatment
53	Stress-rupture curves of SAP 7% at 460°C (thermal cycling during the creep tests)
54	Battelle creep specimen
55	Stress-rupture curve of SAP 4% at 460°C
56	Scatter of strain values of SAP 4% at 460°C
57	Stress-rupture curve of SAP 7% at 460°C
58	Scatter of strain values of SAP 7% at $460^{\circ}$ C
59	AECL creep specimen
60	Stress-rupture curves of three grades of SAP at 426°C (AECL results)
61	Cumulative stress-rupture curves at $460^{\circ}$ C obtained by different laboratories on SAP ISML $4\%$ and $7\%$

## Relaxation Tests

62	ISML relaxation specimen
63	Euratom relaxation specimen
64	Relaxation curves of SAP 7% at $20^{\circ}$ C
65	Relaxation curves of SAP 7 and 10% at high
	temperature
6 <b>5a</b>	Relaxation curves of SAP 7% at 450 $^{\circ}$ C (Euratom
	results)
66	Relaxation curves of SAP 7% at 450°C (ISML results)

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### Impact Strength Tests

67 Charpy test specimen
68 Impact strength as a function of temperature for SAP 4 and 7%

### <u>Hardness</u>

69	Vickers micro- and macrohardness as a function
	of percentage of Al <sub>2</sub> 03 at 20°C
69a	Vickers microhardness of SAP ISML vs. temperature
70	Brinell macrohardness as a function of percentage
	of $Al_2O_3$ at $20^{\circ}C$
70a	Brinell macrohardness of SAP ISML vs temperature
	(Euratom results)
70b	Brinell macrohardness of SAP ISML vs. temperature
	(ISML results)
71	Vickers hardness as a function of the deformation
72	Vickers hardness as a function of annealing tem-
	perature for SAP 4,2 after different deformations
73	Vickers hardness as a function of annealing tem-
	perature for different grades of SAP

#### Fatigue Tests

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74	ISML fatigue specimen
75	Fatigue curves for SAP $4\%$ at 20 and $400^{\circ}$ C
76	Fatigue curves for SAP $7\%$ at 20 and $400^{\circ}$ C
77	Fatigue curves for SAP $10\%$ at 20 and $400^{\circ}$ C
78	Fatigue curves for SAP 14% at 20 and $400^{\circ}$ C
79	Cumulative fatigue curves
80	ISML notched fatigue specimen for $20^{\circ}$ C
81	ISML unnotched fatigue specimen for $20^{\circ}C$
82	Notch effect for SAP 4% at 20 <sup>0</sup> C
83	Notch effect for SAP 7% at 20 <sup>0</sup> C
84	Notch effect for SAP 10% at 20°C
85	Notch effect for SAP 14% at 20°C

Fig.

### Evolution of Rod Production

86	Histograms of mechanical properties of different
	SAF grades at 20 C
87	Histograms of mechanical properties of different
	SAP grades at 400°C
88	Histograms of mechanical properties of SAP 7%
	at $450^{\circ}$ C
89	Evolution of elastic limit $S_{0,2}$ as a function of
	batch number (SAP ISML 7%)
90	Evolution of maximum tensile stress Su as a func-
	tion of batch number (SAP ISML 7%)
91	Evolution of plastic elongation at rupture end
	as a function of batch number (SAP ISML $7\%$ )

### Physical Properties

92	Young's modulus as a function of percentage of
	$Al_{2}0_{3}$ at 20°C
93	Young's modulus as a function of temperature for
	different SAP grades
94	Mean coefficient of thermal expansion as a func-
	tion of percentage of Al <sub>2</sub> 03
95	Electrical resistivity as a function of percentage
	of Al <sub>2</sub> O <sub>3</sub> at 20 <sup>°</sup> C
96	Electrical resistivity as a function of tempera-
	ture for different SAP grades (Euratom results)
97	Electrical resistivity as a function of tempera-
	ture for different SAP grades (ISML results)
98	Electrical resistivity as a function of percentage
	of oxide at 100 and 500°C
99	Inermal conductivity as a function of temperature
	for different grades of SAP
100	Thermal conductivity at 100 and 500°C for different
	grades of SAP
101	Thermal conductivity as a function of temperature
•	for different SAP grades (ISML results)

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102	Thermal conductivity as a function of $\%$ Al <sub>2</sub> O <sub>3</sub> at 500 <sup>°</sup> C
103	Thermal conductivity as a function of $\%$ Al <sub>2</sub> 0 <sub>3</sub> at 100 <sup>°</sup> C
104	Density as a function of $\%$ Al <sub>2</sub> 0 <sub>3</sub> (ISML results)
104A	Density as a function of $\% \text{ Al}_2^{-0}$ (AEK Risö results)

### Finished Products (ISML Results)

105	Tensile tests on smooth tubes as a function of
	percentage of $Al_2O_3$ at 20 and 450°C
106	The elastic limit $\hat{S}_{0.2}$ of smooth tubes as a func- tion of percentage of Al_0_ at 20 and 450°C
107	The plastic breaking strain $e_{pb}$ of smooth tubes as a function of percentage of Al 0 at 20 and $450^{\circ}$
108	Stress-rupture curve for internal gas pressure of smooth tubes of SAP 4% (Euratom results)
109	Stress-rupture curve for internal gas pressure of smooth tubes of SAP 4% (Euratom results)
110	Stress-rupture curve for internal gas pressure of smooth tubes of SAP 7%
111	Stress-rupture curves for internal gas pressure of smooth tubes of SAP 7% obtained by different fabri- cation processes
112	Histograms of mechanical properties $S_u$ and $e_b$ of different finned tubes of SAP ISML 7%
113	Mechanical properties ( $S_u$ and $e_{pb}$ ) of different finned tubes of SAP ISML 7% as a function of batch number
114	Mechanical properties $S_u$ and $e_{pb}$ of tube of SAP 7% (profile ISML-9) as a function of batch number
115	Stress-rupture curves of rod and tube materials (SAP 4%)
116	Stress-rupture curves of rod and tube material (SAP 7%)

<u>Fig</u> .	
117	Histograms of tensile test results on bar and
	tube materials of SAP ISML 7%
118	Burst tests on finned tubes of SAP ISML $7\%$
	(Tangential stress at breaking point vs. depth
	of defect).
119	Fatigue burst tests on finned tubes of SAP ISML $7\%$
	under internal pulsed gas pressure.

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### SEMI - FINISHED PRODUCTS

### TENSILE TESTS





EURATOM TENSILE SPECIMEN TR 1 (gauge length = 7.5 X Diameter)







Fig. 3




























































AECL TENSILE SPECIMEN Ref. 3.3 (Gauge length = 3.9 X diameter)



FIG.31















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ISML CREEP SPECIMEN (Ref. 9)





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Fig. 38





















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Fig. 62

## ISML RELAXATION SPECIMEN (Ref. ISML Drawing P. Dc 27/1)



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Fig. 63













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CHARPY SPECIMEN FOR IMPACT STRENGTH TEST



















VICKERS HARDNESS AT 20°C ON COLD WORKED SAP ISML VS. ANNEALING TEMPERATURE (Euratom resuls Ref. 56)



% Al 203	Load (Kg.)
11,9	2
8,3	1
3,7	2
2.4	1
	% Al <sub>2</sub> 0 <sub>3</sub> 11,9 8,3 3,7 2,4





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d (g/cm<sup>3</sup>) DENSITY OF SAP ISML VS NOMINAL GRADES (ISML Results Ref.64)

FIG.104





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# EXPERIMENTAL CONDITIONS AND COMPLEMENTARY RESULTS



3. EXPERIMENTAL CONDITIONS AND COMPLEMENTARY RESULTS

3.1 Mechanical Properties of Semi-finished products

3.1.1 <u>Tensile Tests</u>

3.1.1.1 Euratom and ISML Laboratory Tests

3.1.1.1.1 <u>Test conditions</u>

#### Test specimen

namely:

First of all, we can analyze the influence of the relation  $x = \frac{\text{length}}{\text{diameter}}$  as related to the specimen, on the elongation value on rupture on SAP ISML 10% for instance. Fig. 1 shows that the elongation decreases with increasing x because the influence of the elongation at necking diminishes with increasing length.

This effect is more clearly visible in Fig. 2, where the elongation values have been related to increasing values of  $\frac{1}{x}$ .

Fig. 2 also shows the values of  $\frac{1}{x}$  for the specimens used by different laboratories (Section 2.1) which studied the SAP ISML,

> Euratom ISML Risö (Ref. 41) AECL (Ref. 33) TLH (Ref. 67 to 86 Part II)

The drawings of the specimens used by Euratom and ISML are given in Figs. 3 and 4 respectively (dimensions in mm).

#### Testing instruments

In the Euratom laboratories we used a universal electric testing machine (Instrom TTCM). The applied load is measured very accurately between 1 g and 5 t by means of a series of load cells, strain gauges being used as transducers. The crosshead speed can be varied very precisely within a range from 0.0005 to 50 cm/min. The influence of the tensile speed on the mechanical properties is presented on diagrams Nos. 6,7,8,9,9A,9B,9C.

In the ISML laboratories a hydraulic testing machine (Amsler) was used. The testing speed varies with the load and cannot be accurately determined. The measurement of the load is obtained directly from the variation of the pressure in the hydraulic system.

#### Measurement of elongation

In most of the current tests no extensometer was used except for very accurate measurements (as in Fig. 10).

In the ISML 4aboratories, which are primarily intended for fabrication control purposes, two marks are engraved on the specimen indicating the gauge length. After rupture the two parts are put together again and the new distance between the two engraved marks measured. For determination of Young's modulus extensometers of the Martens type are used.

In the Euratom laboratories the elongations are determined from the diagrams representing the load (P) as a function of the crosshead displacement ( $\triangle$ L). This latter can be amplified over a wide range; generally, during the elastic part an enlargement of 100 x was used and during the rest of the test 10 x. If we consider the plastic elongation only, then the influence of the frame deflection of the machine is eliminated (Ref. 30).

For special measurements extensometers with inductive transducers or differential methods (specimens of different lengths) are employed.

#### Determination of the engineering yield stress

The engineering yield stress was always determined with the aid of the P =  $f(\Delta L)$  diagrams in continuous tests ( $\Delta L$  was enlarged 100 x). Afterwards a straight line at 0.2% plastic elongation was drawn parallel to the elastic line of proportionality (Ill. 5 & 6). Intermittent tests, causing a plastic elongation of 0.2% after unloading, were therefore not carried out. The diagrams in Fig. 10 give various values for the engineering yield stress, obtained by several methods at 20 and  $450^{\circ}C$  as a function of the percentage of  $Al_{2}O_{3}$  and as a function of temperature for SAP 7%. The engineering yield stress at 0.004% is clearly lower than at 0.2% and is very near the limit of proportionality.

#### 3.1.1.1.2 <u>Results</u>

#### Feature of the load elongation curves

In Illustration 6 are presented fac-simile of load-elongation curves obtained on the Instron recorder. We have chosen two representative tests (on SAP 7% at 20 and  $450^{\circ}$ C) in order to give an idea of the tensile behaviour of SAP material at room and at high temperature. Therefore, the two diagrams have been drawn at the same scale.

It can be seen on these curves that at high temperature, the difference between  $S_{0.2}$  and  $S_u$  is strongly reduced. At the same time, the uniform elongation ( $e_{pu}$ ) decreases till nearly zero, so that the local necking initiates much earlier. In other words the zone of strain-hardening and the plastic reserve have practically disappeared.

<u>Properties as a function of temperature for several grades of SAP</u> For each Al<sub>2</sub>0<sub>3</sub> percentage a diagram of the tensile stresses  $(S_{0.2} \text{ and } S_u)$  and of the tensile strains  $(e_{pu} \text{ and } e_{pb})$  is given as a function of the temperature (20 up to  $600^{\circ}$ C). Each point represents the average of at least three different tests.

Illustration 6 LOAD-ELONGATION CURVES OF SAP ISML 7% Definition of terms applied Stress(S) Load 400 (Kg) (Kg/mm²) 30\_ Su 300 502 20. 200 S=-P 20°C Test 10\_ 100\_ 0. e= AL (%) ерu epb Т 20 0 30 10 Stress(S) (Kg/mm<sup>#</sup>) Load TEST CONDITIONS (Kg) Specimen Fig 3 Crosshead speed: 0.2 cm/mn Magnification of elongations: 25 20\_ 200\_ S=\_\_\_\_\_\_ F. 450℃ Test 10\_ 100 Su Soz 0.1. 0  $e = \frac{\Delta L}{L_*} (*/_*)$ epu epb L > 10 20

#### SAP ISML 4%

The properties were determined on two main batches: one old batch (fabrication of May 1961) and one more recent (fabrication of June 1962) Figs. 11 and 12. This latter batch has slightly better mechanical strength (10% at  $20^{\circ}$ C and 20% at  $450^{\circ}$ C) and lower elongation at  $20^{\circ}$ C.

SAP	ISML	7%	(Figs.	13	and	14)
SAP	ISML	10%	(Figs.	15	and	16)
SAP	ISML	14%	(Figs.	17	and	18)

Observation of the curves:

As a general rule, the tensile stresses and the elongation both decrease with increasing temperature. From about  $300^{\circ}C$ onwards S<sub>u</sub> mingles with S<sub>0.2</sub>, and e<sub>pu</sub> reaches its minimum value which remains nearly constant up to  $600^{\circ}C$ .

Comparison of the four grades of SAP

For each property all four grades of SAP were compared (other presentation of previous results) on the following diagrams:

Property	Fig	5. No.
	Euratom results	IS <b>ML</b> results
Su	19	20
s <sub>0.2</sub>	21	22
e <sub>pu</sub>	23	
e <sub>pb</sub>	24	25

ISML gave averages for the results obtained from production control tests of extruded rods, over a three-year period. The curves are seen to behave similarly for each type of SAP. Cumulative curves as a function of the percentage of Al203

- Diagrams of the tensile stresses (Fig. 26) and elongations (Fig. 27) for various temperatures between 20 and 600°C (values derived from the curves shown in Figs. 11 to 18); The tensile stresses increase with increasing oxide contents. However, the elongations decrease with increasing oxide percentages.
- Curves of the tensile stresses and elon\_ations at 20 and 400°C determined by ISML on extruded rods (Fig. 28).

The latter curves were plotted with the aid of average values taken from the histograms (Figs. 86 and 87), representing three years of fabrication.

#### 3.1.1.2 <u>AECL Laboratory Tests</u> (Ref. 33)

#### Test conditions

The tensile specimens (Fig. 31) were tested on a Hounsfield testing machine and the yield strength determined from the autographic record.

#### Test results

Only the values for SAP 4% are reported here. Tests have been carried out only at room temperature and above  $375^{\circ}$ C. The diagram of the stress versus temperature (Fig. 32) shows approximately the same values as Fig. 11. The diagram of the strain versus temperature (Fig. 33) lacks precision between 20 and  $350^{\circ}$ C but the general form of the  $400-500^{\circ}$ C range is the same as Fig. 12

#### 3.1.1.3 <u>Risö Laboratory Tests</u> (Ref. 41)

#### Test conditions

The tensile specimen has a diameter of 4.5 mm and a gauge length of about 45 mm. Either a 10 t Amsler or a 5 t Instron testing machine was used. With the latter (for more precise tests), a crosshead speed of 0.1 mm/min was applied up to  $S_{0.2}$ , the rest of

test being performed at 1 mm/min. The Amsler or Instron furnaces were regulated to within  $\pm 3^{\circ}$ C and the soaking time before testing was about 30 min. At least two specimens were tested for each temperature and SAP grade. The S<sub>0.2</sub> was always determined continuously with an extensometer.

#### Test results

Fig. 34 gives the properties at 20 and  $400^{\circ}$ C as a function of the percentage of Al<sub>2</sub>O<sub>3</sub>. Compared with the diagrams in Figs. 26 and 27 it will be seen that the tensile stresses agree with the classical values, but the strains are slightly lower, especially at  $400^{\circ}$ C.

## 3.1.2 <u>Compression Tests</u> (Euratom results)

#### 3.1.2.1 Experimental Conditions

#### Shape of the test specimen

We chose the same diameter (4 mm) as for the cylindrical part of the tensile specimen (Fig. 3). After having studied the compression behaviour of specimens from various heights (three times, twice, once the diameter) a cylinder with a height equal to its diameter was taken as a specimen (thus minimizing the effects of parasitic buckling).

#### Testing machine

For the compression test we used an Instron TTCM universal electrical testing machine. The velocity of the crosshead (1mm/min) was enlarged a hundred times on the load-elongation diagram.

#### 3.1.2.2 Behaviour of the "Engineering Stress-Strain" Diagrams

<u>Construction of the curves</u> (Illustration 5, section "Symbols" As for the tensile test, we used the "load-elongation" diagrams plotted by the machine. The section of which the tensile stress can be calculated is the original section of the specimen. The strain is calculated on the basis of the original height of the specimen before the test. Observation of the curves

Ill. 5 shows the behaviour of the diagrams for a tensile test and for a compression test. The symbols used for compression are the same as those for tensile tests (see Table 2), but followed by an apostrophe.

In the elastic region the inclination angles  $\ll$  and  $\ll$ ' of the curves are different, since the deflection of the machine is not the same. This difference is eliminated by considering only the plastic strains. The maximum engineering tensile stress (S'\_u) is assumed to be the stress which corresponds to point B' where the curve shows a point of inflection, this latter also enabling the value of elongation e'\_pu to be calculated. This is logical, whereas in a "true stress/true strain" diagram the tensile and compression curves show the same behaviour, since in that case the point B is also a point of inflection.

3.1.2.3 <u>Results</u>: <u>Cumulative\_curves for tensile and compression</u> <u>stresses\_and\_elongations\_as a function\_of the temperature for</u> <u>the four\_grades of SAP\_ISML</u>

Properties	Results of tensile tests in Fig.	Results of compression tests in Fig.
s' <sub>u</sub>	19	35
s' <sub>0.2</sub>	21	36
e'pu	23	37
e' <sub>pb</sub>	24	

The engineering 0.2 offset yield stress (S' $_{0.2}$ , Fig. 36) under compression is slightly higher than S $_{0.2}$  under tensile stress, especially at 20°C. The maximum engineering compression stress (S' $_{\rm u}$ , Fig. 35) at 20°C is higher than S $_{\rm u}$  (about 30%), but at higher temperatures becomes comparable.

- 44 -

The engineering plastic uniform strains (e'<sub>pu</sub>, Fig. 37) in compression tests are clearly higher than the  $e_{pu}$ -values under tensile stress. The minimum values - which were situated around 300°C for  $e_{pu}$  - are shifted to about 450°C for e'<sub>pu</sub>. Thus this behaviour of e'<sub>pu</sub> looks like that of  $e_{pb}$  under tensile stress (Fig. 23).

In conclusion it may be said that both the tensile and the compression test results show a drop in stress and strain for increasing temperature.

#### 3.1.3 Creep Tests

#### 3.1.3.1 Euratom-ISML Tests

#### 3.1.3.1.1 Test conditions:-

#### - Test\_specimens:

The test specimens used by the Euratom and ISML laboratories are given in Figs. 38 and 39 respectively. The former were taken from rods with a diameter of 9.5mm, and the latter from rods with a diameter of 21 mm.

#### - Testing machines:

#### Euratom tests:

We used Adamel TAC stress-to-rupture machines fitted with a modified electrical accessory permitting magnifications up to 500 times (this magnification value has always been used for tests on SAP because of the low elongation values of this material). The extensometer is fixed above the heads of the test specimens, i.e., at the two ends. The temperature is controlled by two Chromel Alumel thermocouples, and regulation of the furnace keeps the temperature constant to  $\pm 1.5^{\circ}$ C.

#### ISML tests:

The tests quoted in this report were carried out in the laboratories of AIAG/Neuhausen. The extensometer was fixed in two grooves in the heads of the test specimen (Fig. 39). The temperature of the test specimen was maintained at  $\pm 1^{\circ}$ C.

#### 3.1.3.1.2 Stress-to-rupture curves

The ISML tests were performed at  $400^{\circ}$ C and the Euratom tests at 420 and  $460^{\circ}$ C. The stress-to-rupture tests were drafted in semi-logarithmic scales up to 10,000 hr. Each of these curves shows a sudden change in the slope at a point which varies with each percentage of  $Al_2O_3$ . The test specimens show no tertiary creep, and no re-acceleration of the elongation just before rupture of the specimen.

- SAP 4%: Fig. 40
- SAP 7%: Fig. 41: Results for an old batch (made in May 1961). The results at 460°C show a slight scatter.
  - Fig. 42: Stress-to-rupture at 420°C. For the same batch as before, the scatter is lower.
  - Fig. 43: These results refer to more recent batches (1964 and 1966) and show very marked scatter; around a stress of 5 kg/mm<sup>2</sup> the scatter a-mounts to 0.35 kg, i.e., 7%.
  - Fig. 44: Uniform elongations. These values were obtained with the aid of the tests described in Fig. 43 and are tabulated in a histogram. The elongations at loading are of the order of 0.5%. At rupture, the average elongation is of the order of 0.8% and the minimum elongation 0.4%.

<u>SAP 10%:</u> Fig. 45

<u>SAP 14%:</u> Fig. 46

#### 3.1.3.1.3 <u>Cumulative diagrams</u>

The previous results were tabulated in cumulative diagrams as a function of various parameters.

#### Stress-to-rupture curves

- Stress-to-rupture of SAP 7% at 400, 420 and 460°C (Fig.47). The relative position of the curve obtained at 420°C as compared with those at 400 and 460°C does not seem to be very logical when an attempt is made to interpolate linearly for an intermediate temperature.

- Stress-to-rupture at 400°C for the four oxide percentages (ISML results) (Fig. 48). The changing point of the slope for each curve varies somewhat from one oxide percentage to another. The relative curve at 7% seems to be abnormally displaced upwards.
- Stress-to-rupture at 460°C for the four percentages of oxide (Euratom results) (Fig. 49). The curves show rather similar slopes.

Stress to produce creep strain of 0.1% at  $400^{\circ}C$  (ISML results, Fig. 50)

Because there is considerable scatter of the results of the elongation measurements (see histogram in Fig. 44), this curve can only be regarded as a tendency.

Elongation curves at  $400^{\circ}$ C for 1,000 hr as a function of the applied stress (ISML results, Fig. 51)

The values given are very low, but the number of observations is very small, so these curves are merely regarded as indicative.

#### 3.1.3.2 <u>Battelle Institute Tests</u> (Frankfurt)

#### Test conditions

Test specimens (Fig. 54): taken from rods 21 mm in diameter - Testing machine:

The machines used were designed at the Institute. The knives of the extensometer are fixed to the heads (the two ends) of the test specimen. The elongation was measured with the aid of a microscope. The light-beam passed through a quartz window mounted in the wall of the furnace. The absolute accuracy of the measurement is to within about 3 microns. The temperature of the test specimen is kept constant to  $\frac{+}{3}3^{\circ}C$ .

# Stress-to-rupture curves for SAP 4% and 7% at 460°C

#### <u>Stress-to-rupture curves</u> (Fig. 55):

The results refer to two batches (batch 645 of March 1962 and batch 890 of October 1962). For a given stress-to-rupture time,

the scatter of the stress values is of the order of 12%. The values obtained for the more recent batch seem to be lower.

# Elongations (Fig. 56):

The results are so scattered that we preferred to present them in the form of histograms.

Minimum elongation at rupture 0.2 to 0.3%. Average elongation about 0.45%

SAP 7%: The study refers to three batches (December 1961, October 1962, September 1963). Stress-to-rupture (Fig. 57):

The more recent batches seem to be less resistant, especially for long duration.

Elongations (histogram in Fig. 58): Minimum elongation at rupture 0.2 to 0.3% Average elongation about 0.5%

These elongations are comparable to those obtained for SAP ISML 4%.

The elongation scatter has dropped in comparison with that in the Euratom results (Fig. 44) and the mean value of the Battelle data is lower (0.5 as opposed to 0.8%).

#### 3.1.3.3 <u>AECL Tests</u>

# Test specimen (Fig. 59)

# Stress-to-rupture curves of SAP ISML 4, 7 and 10% at $426^{\circ}C$ (800°F) (Fig. 60)

The results obtained by AECL at  $426^{\circ}$ C are as low as those obtained by Euratom at  $460^{\circ}$ C (see Fig. 49). As far as SAP ISML 7% is concerned, these results are 15% less than those obtained by Euratom at  $420^{\circ}$ C (Fig. 47).

# 3.1.3.4 <u>Comparison of Results Obtained by Battelle Institute</u> and <u>Euratom</u>

These two laboratories in fact carried out similar tests on SAP ISML 4% and 7% at  $460^{\circ}$ C. However, the diameter of the rods from

	diameter of rods (mm)	extrusion ratio
rods used by Euratom	9.5	74
rods used by Battelle In- stitute	21	15

which the test specimen were taken and therefore the extrusion ratio were different, as indicated below:-

The stress-to-rupture curves given previously are compared in one diagram (Fig. 61)

#### SAP 4%

The Euratom results are situated in the lower part of the scatter range of the Battelle findings.

#### SAP 7%

The overlapping areas of the results obtained by the two laboratories refer to older batches (No. 551 and 906). The lower values refer to a more recent batch (No. 1300).

#### 3.1.4 <u>Relaxation Tests</u>

3.1.4.1 Test Conditions

#### Test specimen

Test specimen TR 1 (Fig. 3) Test specimen ISML (Fig. 62)

#### Testing machines

#### <u>Electronic\_relaxation machine</u> (Euratom) (Ref. 62)

This machine was developed by Euratom under contract. The test specimen, which is placed in a closed room under vacuum, is fitted with two extensometer shafts connected to a transducer of capacitive displacement. A dynamometer, also connected to a

capacitive transducer, is inserted in the loading system under vacuum. The bridges intended for measuring the load and elongation form a system which is closed in such a way that it is possible to work at constant load (creep test) or at constant elongation (relaxation test), thus eliminating the inherent deformations of the machine frame and the friction forces at the passages into the vacuum chamber. The upper part of the enclosure in which the test specimen is situated is placed in a furnace the temperature of which may be regulated to  $\frac{+}{-}$  1.5°C.

#### Machine for creep test (Adamel TR-3 Euratom) (Ref. 57)

With this apparatus the isothermal viscosity can be tested up to  $60 \text{ kg/mm}^2$  and the stress relaxation up to  $40 \text{ kg/mm}^2$  on a test specimen 4 mm in diameter. The test specimen is put into a furnace (temperature  $\pm 1^{\circ}$ C). The load is applied with the aid of a lever and a spring. The extremities of the two quartz shafts of the extensometer are held in two holes drilled in the axis at the ends of the heads of the specimens. The displacement of the extensometer shafts is mechanically magnified up to 1000 times. The extensometer actuates an electrical contact which starts a motor to change the loading spring.

<u>Relaxation machine of the ISML laboratory</u> (Ref. 21) (Baldwin modified)

Essentially the machine consists of an extensometer which is fixed to the test specimen and sends out the signal for regularization of the load. The variations in the load in time are directly recorded by the machine. The furnace for heating the test specimen is provided with a special regulating system by which the temperature can be kept constant at less than  $1^{\circ}C$ .

<u>Universal testing machine Instron type TTCM</u> (Euratom Ref. 60) The first orientation tests carried out by Euratom were made on this machine, according to a method described by the designer (Ref. 61). Essentially it consists in applying a certain load after which the movable crosshead is held rigid and the decrease in the load as a function of the time is accurately measured. The application of this method is rather tricky because the deflection of the machine frame and the temperature variations have to be taken into consideration.

A correction factor for the machine deflection could be established approximately from a short test series in the following way. An extensometer was used (magnification 1000 x and an error of less than 1%); after the desired initial load had been applied, the position of the crosshead was corrected manually in order to maintain the initial elongation of the specimen.

The decrease in the load as a function of the time thus measured was three times as high as the values found earlier by the simpler method. We therefore applied this correction factor, equal to 3, in the following results.

#### 3.1.4.2 Euratom Results

We principally studied the SAP ISML 7% and performed three series of tests, applying initial stresses equal to 1/3, 2/3 and 3/3 of the yield point at 0.2% ( $S_{0,2}$ ).

# <u>at 20<sup>0</sup>C</u> (Refs. 58-60, Fig. 64)

The preliminary results give an idea of the behaviour of the curves. The decrease in the stress expressed as a percentage of the initial stress is indicated in the table below:-

	Decrease in the stress %					
Initial stress	1 hr	5 hr	10 hr	100 hr		
1/3 S <sub>0.2</sub>	3	5	_	_		
2/3 S <sub>0.2</sub>	5	9	11	-		
3/3 S <sub>0.2</sub>	18	23	-	_		

# <u>at 450°C</u> (Refs. 57, 58 and 60, Fig. 65)

For stresses equal to 2/3 of the yield point, the curves show a change in slope which is especially visible in the region of 1 hr.

For lower stresses, the slope of the curve is less. The slopes of the curves obtained with three different machines are very comparable. The decrease in the stresses is reported in the table below:-

In Fig. 65a only the relaxation curves obtained with the Instron machine are shown. They correspond to initial stresses of 1/3, 2/3 and 3/3 of S<sub>0.2</sub> at  $450^{\circ}$ C. The higher the stress, the steeper the slope.

	Decrease in the stress $\%$			
Initial stress	1 hr	10 hr	100 hr	
1/3 S <sub>0.2</sub>	16	30	40	
2/3 S <sub>0.2</sub>	20	40	70	
3/3 S <sub>0.2</sub>	ruptured after 15 min.			

3.1.4.3 ISML Results (Ref. 21, Fig. 66)

The partial results given here are for SAP 7%. Preliminary creep tests have shown that the deformation imposed at the beginning of the test should not subject the material to stresses in excess of 5 kg/mm<sup>2</sup> (i.e., 2/3 of the yield point). The first test specimen underwent a deformation of 0.165%.

The decrease in the stress is reported in the table below:-

Initial stress	Decrease in the stress $\%$			
	1 hr	10 hr	100 hr	
0.55 S <sub>0.2</sub>	25	40	45	

The behaviour of the curve is very different from that obtained on the various machines used by Euratom. The phenomenon seems to slow down after 10 hr, whereas in the other cases it speeds up.

#### 3.1.5 Impact Strength Tests (Refs. 9 and 10)

#### 3.1.5.1 Test Conditions

The standard Charpy specimen was used (Fig. 67). The specimen was machined from bar stock 10.3 x 10.3 mm square (E = 60) and afterwards annealed for two hours at  $500^{\circ}$ C. The instrument used was an Amsler machine; the hammer energy applied was 30 mkg. With the same hammer lower energies were applied, as indicated in Fig. 68. For the high-temperature tests every specimen was soaked at its testing temperature for 30 min. Generally five tests were performed for each temperature.

#### 3.1.5.2 <u>Results</u>

The impact strength of SAP ISML 4% and 7% Al<sub>2</sub>O<sub>3</sub> versus temperature is shown in Fig. 68. It is clear that the impact strength increases with temperature. The part of the curve shown as a dotted line indicates values which are too low because at these points the specimens did not rupture completely.

The curve for SAP 7% lies below the 4% Al<sub>2</sub>O<sub>3</sub> curve, thus indicating a lower ductility, which is in agreement with other types of mechanical tests. There are two individual points (at 20 and  $400^{\circ}$ C) for SAP 4% which are well below the established SAP 4%curve. These points were obtained for a very old batch made under quite different fabrication conditions (only simple extrusion was applied).

#### 3.1.6 <u>Hardness Tests</u>

#### 3.1.6.1 <u>Macrohardness</u>

Brinell Macrohardness: ISML (Ref. 2-4) measured the Brinell macrohardness at 20°C applying a ball diameter of 2.5 mm, a load of 62.5 kg and a time of 30 secs. Results as a function of oxide contents are given on Fig. 70.

The same laboratory performed tests up to  $400^{\circ}$ C on SAP 7 and  $14_{7'}$  (Ref. 21b). The test conditions were as follows: ball diameter 5 mm, load 125 kg maintained during 3 min. Results are given on Fig. 70b.

Similar tests have been done in Euratom laboratories up to 600°C with a machine of Euratom construction. The test conditions were as follows:-

- ball diameter: 2.5 mm
- loading speed: 0.4 mm/s
- load maintained during 3 mins.
- number of indentations for each temperature: 4
- maximum difference of temperature between two indentations: ± 3°C.

Results are reported on Fig. 70 a

<u>Vickers Macrohardness</u>: Euratom measured the Macrohardness with a Tukon-Wilson tester by the Vickers method. A load of 1 or 2 kg was used as can be seen in Fig. 69. Five different samples were prepared for each SAP grade and three separate indentations were made on each sample.

#### 3.1.6.2 <u>Vickers Microhardness</u> (Euratom results)

Euratom measured the microhardness at room temperature with a Leitz-Durimet by the Vickers method (load 100 g, time 10 s). The arithmetic mean of ten measurements for each grade of SAP is reported in Fig. 69.

Preliminary results of Vickers Microhardness at high temperature have also been obtained in the Euratom laboratories with a new machine developed under contract (S.E.A.V.O.M./EURATOM)(Ref. 28bis). The maximum difference of temperature between two successive indentations on the same specimen is reported on the table below:-

Test temperature <sup>O</sup> C	Maximum temperature difference
1,000	+ 8
600	+ 4
400	+ 2

The indenters are made of diamond. The heating of test specimen is obtained through electron bombardment, the vacuum of the chamber being maintained between  $10^{-5}$  and  $10^{-6}$  Torr.

The test conditions were as follows:

- load: 50 g
- loading duration: 1s
- load maintained during 20 s
- number of indentations for each temperature: 3 to 4
- number of measurements possible on one specimen: >100
- the measurement of indentation is made at the required temperature

The results are given on Fig. 69A.

#### 3.1.7 Fatigue Tests by Rotating Bending (ISML results)

# 3.1.7.1 Fatigue Life at $20^{\circ}$ and $400^{\circ}C$

Test conditions (Ref. 8)

The fatigue test specimen for this series of tests is shown in Fig. 74. The surface finish was held between 0.09 and 0.1 micron and was measured by a Taylor-Hobson roughness meter in the longitudinal direction of the specimen. The rotating bending fatigue machine was a Schenck-Duplex, turning at 3,000 rpm.

#### Results

The results are shown in Figs. 75-79 for 20 and  $400^{\circ}$ C. Generally speaking, the  $400^{\circ}$ C curve is about 50% lower than the  $20^{\circ}$ C curve. The maximum number of cycles was  $10^{8}$ , but the curves did not reach the asymptotic value at this point, so the real fatigue limit is not known. Thus we can only speak of a fatigue life at  $10^{8}$  cycles.

From the same batch some tensile tests were carried out in order to compare the results of the two tests.

Ratio % Al <sub>2</sub> 0 <sub>3</sub> <u>F</u> S <sub>0.2</sub>	4	'7	10	14
at 20 <sup>0</sup> C	0.56	0.48	0.49	0.49
at 400 <sup>0</sup> C	0.92	0.71	0.68	0.60

In the above table the ratio between the fatigue life at  $10^8$  cycles (F) and the 0.2 offset yield stress is given for four SAP grades at 20 and  $400^9$ C.

# 3.1.7.2 <u>Notch Effect in Fatigue Tests by Rotating Bending at 20<sup>°</sup>C</u> <u>Test conditions</u> (Ref. 2)

The unnotched specimen is shown in Fig. 81 and the notched one in Fig. 80. The details of the V- and U-notch are also visible in Fig. 80. The surface finish was held between 0.09 and 0.1 micron, as above. The apparatus was an ISML FRS type turning at 11,500 rpm.

#### Results

The results are given in Figs. 82-85. Each graph shows the unnotched and notched (U-form) fatigue results up to  $10^8$  cycles for a given SAP alloy (4,7, 10 or 14% Al<sub>2</sub>0<sub>3</sub>).

Ratio at 20°C	4	7	10	14
$\frac{\frac{F_n}{n}}{F}$	0.42	0.48	0.45	0.55

The ratio of the fatigue life (Fn) up to  $10^8$  cycles for the notched (U-form) and unnotched specimen (F) is given above. Generally speaking, the notch effect is less severe with increasing  $Al_2O_3$  contents. Besides the U-notch (Neuber coefficient = 2.2), a V-notch (Neuber coefficient = 9) was applied in two test series on SAP 4% and 7%.

From Figs. 82 and 83 it can be seen that the form of the notch has no major influence, except for low cycle values of the SAP 4%.

With different conditions, as in Section 3.1.7.1, we can again determine the ratio  $F/S_{0.2}$  at 20<sup>o</sup>C for the unnotched specimen.

Ratio at 20°C	4	7	10	14
$\frac{F_n}{S_{0.2}}$	0.60	0.50	0.55	0.45

The ratio between the fatigue life (Fn) at  $10^8$  cycles and the 0.2 offset tensile yield stress is given in the above table. The same tendency is visible as in Section 3.1.7.1, that is to say, the reduction in the strain (difference  $S_{0.2}$  - Fn) is more severe for a high percentage of  $Al_2O_3$ .

3.1.8 Gradual Evolution of Batch Quality (ISML Results)

3.1.8.1 Scatter of Results (Refs. 11 and 35)

Histograms of the tensile properties ( $S_{0.2} - S_u - e_{pb}$ ) of different grades of SAP are given in Figs. 86-87-88.

They originate from:-

- a) production controls of the standardized production
- b) controls of major changes in the standardized production method
- c) verification of batches destined for special laboratory test series (e.g., impact strength, creep,fatigue, hardness, etc.).

In Figs. 86 and 87, which give the results for 20 and 400°C, the data are not complete because the numbers in the ordinates of the histograms are not listed. Furthermore, the results in Figs. 86 and 87 are for very old batches produced before the end of 1962.

The more recent results on complete histograms are given in Fig. 88, which contains 43 test results for SAP 7% at  $450^{\circ}$ C.

These 43 tensile tests were selected from batches which were fabricated under standard conditions. For the histograms of Fig. 88 the scatter can be roughly defined as:

for the stresses  $\frac{8 \cdot 1 - 6 \cdot 8}{7 \cdot 57} = 17\%$ <br/>for the strains  $\frac{6 - 2 \cdot 5}{4 \cdot 38} = 80\%$ 

3.1.8.2 <u>Properties as a Function of Batch Number</u> (Ref. 35) Figs. 89-91 showed the curves of the mechanical properties  $(S_{0.2} - S_u - e_{pb})$ .

The tensile test results were selected in order to represent only batches made under standard fabrication conditions.

The method of manufacturing the base material (the starting powder) was unchanged until the end of 1962 (batch No. 1,000). Furthermore, the tensile testing temperature was changed from 400 to  $450^{\circ}$ C.

The three diagrams give an idea of the evolution. There is instantaneous scatter - in the vertical sense, as for batch numbers which are close to one another - and long-term scatter in the horizontal sense, as with the time axis. These two types of scatter are difficult to express in figures owing to the lack of results.

The existence of this "scatter range" also has an effect on the 85 preceding diagrams. These are results relating to a certain batch number and thus these data are not representative for SAP as a whole.

If the results of the previous 85 diagrams are to be used, it will be necessary to apply a safety margin, which is related to the width of the "scatter range".

# 3.2 <u>Influence of the Mechanical and Thermal Treatments on the</u> <u>Extruded Product</u>.

The extruded product is only the base material for the preparation of the finished products. It has already been stated (Section 1.7) that in certain cases (thin-walled tubes, plates, foils) it is necessary to use cold-deformation (rolling or cold-drawing). The evolution of the mechanical properties after cold-deformation as compared with the properties of the extruded product was therefore studied. Furthermore, it is nearly always necessary to include an annealing treatment and often the operating temperature of the final product is quite high, so the evolution of the properties of the material as a function of the annealingtemperature was examined.

The main results obtained are given here and the reader is referred to the specific paragraphs of each type of test for details of the general experimental conditions.

#### 3.2.1 Influence of Cold-Deformation

3.2.1.1 <u>Tensile Tests</u> (Refs. 27 and 46, Figs. 28 and 29) The study was carried out on SAP ISML 4% extruded (E = 2.5) and then rolled (H = 92%). At room temperature the rupture strength of the rolled product (Fig. 28) is higher than that of the extruded material. At  $20^{\circ}$ C, as expected, the plastic strain (Fig. 29) is lower after cold-deformation. The high-temperature tensile tests (heating time + time at temperature 1 hour, so annealing effect cannot be avoided) demonstrate lower strength and higher ductility in the 300-500°C range for the cold-deformed samples. Near 600°C, however, the differences become negligible.

#### 3.2.1.2 <u>Vickers hardness tests</u> (Ref. 56)

The Vickers hardness versus the degree of deformation for aluminium and the four SAP ISML alloys is plotted in Fig. 71. For the same degree of deformation the absolute increase in the Vickers hardness rises with the  $Al_2O_3$  content.

#### 3.2.2 Influence of Heat Treatment After Rolling

3.2.2.1 Tensile Test (Refs. 27 and 46, Figs. 28 and 29)

The tests were performed on SAP ISML 4% only. With a treatment of 3 hr at  $620^{\circ}$ C an attempt was made to recrystallize the rolled material, or at least to recover it.

In diagrams (Figs. 28 and 29) this recrystallization effect is clearly noted for tensile tests at  $20^{\circ}$ C, that is to say, for increasing ductility and decreasing strength values.

Some strange phenomenon occurs at 300 and  $400^{\circ}$ C. At  $300^{\circ}$ C, the ductility of the recrystallized material has the same ductility values as the extruded material. At  $400^{\circ}$ C the strength of the recrystallized material lies between the rolled and the extruded strengths.

# 3.2.2.2 Vickers Hardness (Ref. 56)

In Fig. 72 the recovery of Vickers hardness from H  $_{
m v}$  75 to H  $_{
m v}$  52 is plotted versus the annealing temperature for SAP ISML 4%. All the specimens were annealed together for one hour at the temporatures given in the figures. The specimens were alternately heat-treated, then measured, etc., right through the whole temperature range from 20 to 660°C. This figure shows that for an alloy which has undergone only 22% deformation complote recovery is obtained only at extremely high annealing temperatures. With increasing deformation, the recovery temperature is shifted to a slightly lower temperature. Fig. 73 shows the change in the recovery temperature of different alloys for a deformation of 95%. A shift in the recovery temperature to higher values for increasing Al<sub>2</sub>0<sub>5</sub> contents is clearly visible. In conclusion, it may be said that the SAP structure is stable, recovery and in particular recrystallization being obtained only by thermomechanical treatment which lay completely outside the field of industrial practice.

# 3.2.3 Influence of the Heat Treatment on SAP 45 at 460°C (Ref. 48)

Creep tests were carried out on test specimens which were taken from the same batch and had undergone heat treatment at extremely high temperature (540°C; melting point of Al = 660°C) for periods of between several hours and 3,000 hr. The results are given in Fig. 52. It can be seen that treatment at high temperature of very long duration considerably lowers the properties of the material.

# 3.2.4 Influence of Thermal Cycling During Creep Test (Ref.49, Fig. 53

Thermal cycling of the specimen was effected in the course of a creep test at  $460^{\circ}$ C by switching off the heating furnace four times in 24 hr until the temperature reached  $340^{\circ}$ C. No influence on the stress-to-rupture curve was observed.

## 3.2.5 Influence of previous creep test on tensile properties

## 3.2.5.1 <u>Euratom results</u>

In order to see if the elongation of the specimen in creep test  $(at 460^{\circ}C)$  does reduce the amount of elongation obtainable in tensile tests (at 450°C) we performed tensile tests on specimens which had remained unbroken after creep tests at a stress level of 0.6 Su. The test conditions, both for creep and tensile tests were the standard ones (see Sections 3.1.1.1 and 3.1.3.1) but naturally the creep specimen (Fig. 38) was also used for tensile tests.

Tests have been done on an old Batch (No. 275) and a more recent one (No. 1720-1722). The final results are given on the table below.

> Evolution of tensile properties after creep of SAP ISML 7% related to tensile properties without previous creep

Material: SAP 7%		Evolution of tensile properties			
Batch No.	Origin of powder	$\Delta S_{0.2}$ (kg/mm <sup>2</sup> )	$\Delta S_u^{(kg/mm^2)}$	∆e <sub>pu</sub> %	∆e <sub>pb</sub> %
275	AIAG	- 3%	- 2.7%	-27.3%	-13.5%
1720 1722	Eckart-Werke	+4.2%	+ 3.2%	-40%	-45.3%

The behaviour of the two batches are opposite in strength. However for both batches, the elongation shows a tremendous decrease.

#### 3.2.5.2 <u>ISML results</u> (Ref. 14)

Tests have been done at  $400^{\circ}$ C on old batches of SAP ISML 4, 10 and 14%. The test specimens (Fig. 39) machined from bars Ø 21 mm have been submitted to creep tests up to 5,000 hours at a stress level of 0.66 S<sub>11</sub>, without rupture. Tensile specimens (Fig. 4) were machined out of creep specimens and then tested. As for Euratom results, we give on the table below the evolution of tensile properties.

Material		Evolution of tensile properties			
Batch No.	Origin of powder	A1203	$\Delta s_u (kg/mm^2)$	$\Delta e_{pb}$ (kg/mm <sup>2</sup> )	
381	AIAG	4%	+5.7%	-30%	
409	11	10%	-6.0%	-57%	
416	11	14%	-5.0%	-56%	

As a comparison basis, we have chosen the values of tensile properties obtained on bars  $\emptyset$  16 mm according to Fig. 27. We had previously shown (Fig. 24) that there was no large increase in the values of e<sub>pb</sub> from a bar  $\emptyset$  16 and a bar  $\emptyset$  21 mm.

We see that the decrease of elongation is in good agreement with Euratom conclusions.

#### 3.3. Mechanical properties of finished products

#### 3.3.1 Smooth Tubes

3.3.1.1 <u>Tensile Tests</u> (ISML Results, Ref. 22)

Figs. 105 to 107 contain some tensile properties  $(S_{0.2}-S_u-e_{pb})$  as a function of the percentage of  $Al_2O_3$  for:-

- simple extruded smooth tubes (position 2)
- extruded smooth tubes, afterwards cold-drawn (position 3) Other test conditions are not known.

As was already known in the case of bar material, it will be seen that an increase in the  $Al_2O_3$  content is accompanied by increasing stress and decreasing strain. If we compare positions 2 and 3 in Figs. 105, 107 and 108, the influence of the cold deformation can clearly be seen:

- at 20°C higher stresses and lower strains
- at 450°C lower stresses (especially for SAP 10%) and no significant differences in strain.

### 3.3.1.2 Burst Tests (Euratom Results)

The tubes were tested in a furnace at  $460^{\circ}$ C under internal gaspressure (Nitrogen) up to rupture. The test specimens were 300 mm long and a thermocouple was set inside the test specimen. They were closed by two plugs consisting of an internal aluminium ring pressed on to the tube wall by two cones. The rate of pressure increase was about 10 kg/cm<sup>2</sup> per minute, so the test duration was comprised between 4 and 7 minutes.

The smooth tubes were extruded and then cold-drawn but not annealed. They were left in furnace  $1^{1}/2$  hours for temperature equilibration. The resulting transverse stresses (hoop-stress) (in kg/mm<sup>2</sup>) are listed in the following table.

	% Al <sub>2</sub> 03	4	7		
Test	Batch No.	Production early 1962	2265 to 2273		
specimen	diameters (mm)	13.1 14.7	25.5 27.5		
Test conditions	Duration (min)	7	4		
	Number of tests	5	18		
Results (tangential rupture stress in kg/mm <sup>2</sup> )	Maximum	4.3	5.9		
	Average	4.0	5.2		
	Minimum	3.5	4.1		
Scatter %		20%	35%		

Referring to tensile tests on smooth tubes (Fig. 105), we find:

SAP 4%  $S_u = 5 \text{ kg/mm}^2$ SAP 7%  $S_u = 6.3 \text{ kg/mm}^2$ 

so the ratio for transverse stress (S tr) and longitudinal stress (S lo) becomes

for SAP 4%  $S_{10} / S_{tr} = \frac{5.0}{4.0} = 1.25$ for SAP 7%  $S_{10} / S_{tr} = \frac{6.3}{5.2} = 1.2$ 

However, the thermal treatment applied to burst test specimens and to tensile test specimens are not the same, so, the difference in transversal and longitudinal rupture stresses would be somewhat higher.

# 3.3.1.3 <u>Stress-rupture Tests Under Internal Gas Pressure</u> (Euratom and ISML results) Refs. 8 and 52.

Both laboratories used the same principle as described above in Section 3.3.1.2, but for these creep tests it was necessary to maintain a certain constant gas pressure and to record the timeto-rupture.

Stress rupture curves up to 100 hours are given for SAP 4% and 7% and 400 and 450°C in Figs. 108 and 110. The influence of the temperature and  $Al_2O_3$  percentage are quite normal. A more complete curve up to about 1,500 hours based on two different batches of SAP 4% is given in Fig. 109.

A comparison between two different laboratories (and different batches) is made in Fig. 115. The Euratom results at 100 hours are about 25% lower than those of ISML.

The creep properties of three different production processes are compared in Fig. 111. The creep properties of the extruded tube are largely superior to the tubes which are afterwards colddrawn and the stresses drop again when the tubes are annealed afterwards (maximum difference about 50%).

#### 3.3.2 Finned Tubes

ISML fabricated different types of finned tubes necessary for the different solutions of neutron - and heat-transfer - calculations. The general features of the tube profiles are given in the table below.

Profile number Description	3	6	7	8	9	10	11	12
No. of thermal fins	36	36	36	32	36	36	36	28
No. of spacers	3	3	6	2	3	3	3	-
type of fins straight S helical H	Η	H	S	H	H	Н	S	Η
internal diameter (mm)	25.5	25.5	25.5	21.15	25.4	24.9	24.9	14.6
wall-thickness (mm) tolerances + 0.2 - 0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	0.9
external diameter of thermal fins	29.9	29.5	29.5	24.95	29.8	28.9	28.9	18.4
external diameter of spacers	33.5	30.5	30.5	27.55	32.1	29.9	29.9	_
	A		A			1		

#### 3.3.2.1 Tensile Tests

Many tensile tests were performed for production control purposes. Two properties ( $S_u$  and  $e_{pb}$ ) are shown in histograms in Fig. 112 in order to give an idea of the scatter. Two types of histograms are superimposed, one for the results for all the tubes, and the other for profile No. 9. As usual, the elongations are measured after rupture by putting the two parts of the tube together. The new distance between two marks is then determined. However, from this value must be subtracted the maximum clearance which remains when the two halves are put together.

The arithmetical mean values of profile No. 9 are about 2% higher than the values for all the tubes, so this single profile seems to be really representative of the whole bulk. Comparison between extruded finned tubes and smooth tubes shows good agreement.

properties at 450°C for 7½ SAP types of specimen	S <sub>u</sub> (Kg/mm <sup>2</sup> )	epb(%)
smooth tubes (annealed 2 hr/500°C: Fig. 105 Ref. 22-31)	8.6	2.3 to 3.7
finned tubes (non-annealed)	8.9	2.9
finned tubes profile No. 9 (non-annealed) Fig. 112, Ref. 35	9.2	3.3

### 3.3.2.2 Compression tests

#### Experimental conditions

As for cylindrical specimens, an Instron TTOML testing machine was used and the tests carried out at 450°C. The test specimens consisted of pieces of tube carefully cut perpendicular to their axis. Their length was approximately equal to the greater external diameter of the tube.

#### Results

The rupture stresses are the ratio of the rupture load and of the cross-area (S) of the tube. The "maximum engineering stress", the "engineering 0.2 offset yield stress" and the "plastic uniform strain" are defined as for cylindrical specimens (see Illustration 5). Here, the values of  $e_{\rm pb}$  have no sense, because the breaking point is not reached, as it is in tension tests. The main results are listed on the following table (mean value, max. and min. value).

Characteristics of the Specimen				Results		
Profile of the tube	Height (mm)	Cross area (mm <sup>-</sup> )	Number of tests	S'u (kg/mm <sup>2</sup> )	S'0.2 (kg/mm <sup>2</sup> )	e'pu
ISML 9	30	146	18	7.26 7.15 to 7.39	5.34 4.93 to 5.61	0.32 0.20 to 0.39
ISML 12	18	72	12	6.90 6.86 to 7.0	5.44 5.0 to 5.83	0.20 0.16 to 0.26
Profile	Type of Test					
------------------	----------------	------	-----------------------	------		
of the tube	Tension (ISML)		Compression (Euratom)			
	Su	epb	s' <sub>u</sub>	e'pu		
ISML 9	17%	118%	3%	60%		
ISML 12			2%	50%		
All finned tubes	130	166%				

A rough estimation of the scatter (as in Section 3.9.1) for tensile and compression tests is given in the following table.

We notice that the scatter is always lower in compression. Moreover this last type of test is easier to perform for production control and allows the values of elongation to be obtained more precisely, which lowers the scatter.

## 3.3.2.3 Burst tests

<u>Burst tests as production control</u> (Euratom results)

Sixty burst tests have been done on two main profiles in SAP 7%. The test conditions are similar to those for smooth tubes (see Section 3.3.1.2). For the calculation of tangential stress, the thin-wall formula  $\mathfrak{S}_{t} = \frac{P \cdot Di}{2 \cdot t}$  was consistently used, "t" representing the minimum wall thickness (i.e. without fins) of the tube considering a medium tolerance. The results are assembled on the following table (mean value, max. and min. value).

Specimen		Results			
Profile of the tube	Number of specimens	Burst pressure kg/cm <sup>2</sup>	Tangential rupture stress <b>G.</b> t(kg/mm <sup>2</sup> )	$scatter \ \%$	
ISML 9	30	48 (44 to 50.5)	5.55 (5.1 to 5.8)	12.5	
ISML 12	30	69 (64 to 74)	5.6 (5.2 to 6.0)	14	

- 67 -

Most ruptures occurred at the bottom and along one of the higher spacer fins. The comparison between smooth entruded and cold drawn tubes and finned extruded tubes of SAP 7% is reported in the table below.

Profile of the tube	Number of specimens	Tangential rupture stress 6.t (kg/mm <sup>2</sup> )	Scatter %
Smooth tube	13	5.2	35%
ISML 9	30	5.55	12.5%
ISML 12	30	5.6	14%

The values of rupture stresses for finned tubes are about 7% higher and the scatter is only the half.

The ratio between tangential and longitudinal stresses in finned tubes as compared with smooth tubes is given below.

Type of tube	Tension test	Burst test	Ratio
Smooth	6.3	5.2	1.2
Finned	9	5.6	1.6

This result shows that in finned tubes the difference in mechanical strength in the two directions is higher than in smooth tubes.

# 3.3.2.4 <u>Influence of internal artificial defects on the transverse</u> mechanical properties of finned tubes.

Preliminary studies have been carried out at 450°C on finned tubes (ISML 9) with internal artificial defects machined with a constant profile tool (Ref. 62 bis). All tubes were first ultrasonic tested to ensure that they were free from any natural defect and their thickness bet een every fin carefully measured. The form and dimension of the defects are shown on Illustration 7. Its location is represented on Position 3. A macrography shows on Illustration 8 the real profile obtained. Ten specimens have



Shape and dimensions of the defect











<u>Illustration 8</u> Artificial defect 0.15 mm depth machined on finned tubes (magnification 50) been tested for each type of defect. The results are summarized in the table below. The tangential stress is calculated using the wall thickness measured on the ruptured generatrix: Most ruptures occur along and at the bottom of a spacer fin; only 30% of all the tubes ruptured on the defect.

Depth of defect (mm)	Mean value of tangen- tial stress (kg/mm <sup>2</sup> )	Seatter (%)	Decrease of strength (%)
0	5.52	6.5%	0
0.05	5.45	8.5%	1.3%
0.10	5.40	10.5%	2.4%
0.15	5.34	12 %	3.3%

We notice that the scatter increases with increasing the depth of the defect, but remains lower than in production control tests. The mean values of the rupture stresses show a slight decrease of only 3.3% which is of no real significance due to the large scatter.

# 3.3.2.5 Fatigue tests under internal gas pressure

#### Experimental conditions

The test arrangement is similar to the one used for smooth tubes (see Section 5.1.2). Experiments have been carried out on test specimens 300 mm length of finned tubes profile ISML 9 free from natural defects (U.S. controlled) but having artificial internal defects as described in the previous section. The pressure rose from 1 kg/cm<sup>2</sup> up to the preselected value in approximately 1 minute and was decreased to the initial value nearly instantaneously.

#### Results

As previously, the tangential rupture stress is calculated with the classical  $\mathcal{O}_{t} = \frac{P \cdot Di}{2 \cdot t}$  formula, "t" being the thickness along the line of rupture. As a matter of fact, 66% of all the

tubes ruptured outside the zone of the artificial defects. Most ruptures occurred at the bottom and along one of the three spacer fins. The results are reported on the diagram Fig. 119. We see clearly that the rupture stresses of tubes with defects of 0.05 and 0.10 mm and the most part of those of 0.15 are contained in the dispersion band of the values for tubes without defects. Such short longitudinal defects affecting 10% of the wall thickness do not seem to be very critical even in fatigue testing.

#### 3.3.2.6 <u>Creep tests in compression</u> (Euratom results)

## Test conditions

We used the Adamel TAC stress to rupture machines (as in Section 3.4.1.1) fitted with a cage system allowing work in compression. The test temperature was  $460^{\circ}$ C. Specimens were pieces of ISML 7 finned tubes of 40 mm length (cross section 138 mm<sup>2</sup>).

Results:The number of specimens tested at each stressis as follows $3.8 \text{ kg/mm}^2$  $3.8 \text{ kg/mm}^2$ 3 tests $4.0 \text{ kg/mm}^2$ 3 tests $5.0 \text{ kg/mm}^2$ 6 tests

In these conditions only three specimens at 5.0 kg ruptured after 21, 817 and 2,089 hours respectively, all the others remaining unbroken after more than 4,000 hours. Similar experiments made on cylinders have not ruptured even under a stress of 5.0 kg/mm<sup>2</sup>.

#### 3.3.2.7 Gradual Evolution of Batch Quality

As for the bar production (see Figs. 89-91) we have plotted the tensile properties as a function of the batch number.

Fig. 113 concerns all the tubes as a whole and Fig. 114 profile No. 9 only.

A scatter range is clearly visible in all cases. However, over a long period there is a general tendency for the properties to increase.

#### 3.3.3 Pressure Tubes

The main results obtained are given in Ref. 55.

#### 3.4 Comparison Between Rods and Tubes

#### 3.4.1 Smooth Tubes

#### Creep tests

The creep properties of SAP 4% and 7% are compared in Figs. 115 and 116 respectively. The critical stresses act in two different directions, relative to the fibrous  $Al_00_3$  structure.

Thus, for rod material it is a longitudinal stress and for tube material (creep test with internal gas pressure) transverse. The following table for smooth tubes and rods can be drawn up for the ratio between these two stresses after 100 hr creeprupture.

ratio	Slow testing (creep)	Fast testing (tensile & burst tests) (Section 5.1.2 and 5.2.3)
4	$s_{10}/s_{tr} = 1.3 to_{2.1}$	$s_{10}/s_{tr} = 1.25$
- 7	$s_{10}/s_{tr} = 1.35$	$S_{lo}/S_{tr} = 1.2$

This ratio is compared to the fast testing ratio. It will be seen that the difference between the transverse and longitudinal strengths is greater during slow testing.

### Tension tests

In Figs. 105-107 the properties of smooth tubes and extruded rods (position 1). are compared.

Generally speaking, it may be said that for smooth tubes the strains are lower for 20 and  $450^{\circ}$ C and the stresses also for the  $450^{\circ}$ C tests.

#### 3.4.2 Finned tubes

Histograms of tensile tests of tubes and rods of SAP ISML 7% at  $450^{\circ}$ C are given in Fig. 117.

	Properties at 450°C		
Specimen	S <sub>u</sub> (kg/mm <sup>2</sup> )	e <sub>pb</sub> 🖗	
rod	7.6	4•4	
tube	9.0	3.2	

Note the higher strength (18%) and lower ductility (a decrease of 27\%) of the tubes.

#### 3.5 Physical Proverties

Since the aim of this report is mainly to describe the mechanical properties, a complete description of the physical properties will not be given. We shall limit ourselves to those which could be of immediate interest to the designer.

3.5.1 Young's Modulus
3.5.1.1 Experimental devices
3.5.1.1.1 Static methods

Euratom tests:

All these tests were carried out on a universal electrical Instron machine.

Tests at 20<sup>°</sup>C (Ref. 27, Fig. 92).

Test specimen diameter 4 mm, gauge length 25.4 mm, drawn from bars of 9.5 mm, extrusion ratio E = 76.

Elongation measured with the aid of a Wideman-Baldwin inductive extensometer type PSH 8 MS (magnification 500 x). The results were taken from diagrams plotted by the xy recorder of the Instron machine (load as a function of specimen elongation).

High temperature tests (Ref. 30, Fig. 93)

Test specimens: 1st test specimen (Fig. 3) diameter 4 mm, length 30 mm. 2nd test specimen diameter 4 mm, length 5 mm. We used a differential method involving the use of two test specimens of different lengths so as to eliminate the effects are to the deflection of the frame of the machine. The furnace kept the temperature constant at  $\pm 1^{\circ}$ C.

ISML tests (Ref. 6).

This laboratory used test specimens 10 mm in diameter and 100 mm long drawn from bars 16 mm in diameter (extrusion ratio E = 26.6). The testing machine was an Amsler hydraulic machine. The elongations were measured with the aid of a Martens-type extensometer (magnification 500 x). The furnace was set to  $\pm 2^{\circ}$ C.

3.5.1.1.2 Dynamic methods

Euratom tests (Ref. 29, Fig. 92)

These were performed by internal damping on a Bordoni-type machine (Ref. 24) designed by Euratom.

ISAL tests (Ref. 3, Fig. 92)

The apparatus used was of the Bordoni type (Ref. 24) with electrostatic encitation. The test specimens were cylinders 8 mm in diameter and 160 mm long obtained by double extrusion (extrusion ratio E = 107). The measurements were made under a vacuum of 1.5 mm Hg. The damping of the oscillations was observed with the aid of an oscilloscope.

3.5.1.2 <u>Results</u> 3.5.1.2.1 <u>Young's modulus at 20<sup>°</sup>C as a function of the oxide</u> <u>content</u> (Fig. 92)

Young's modulus increases with the oxide content.

Static method:

- Euratom tests Each point represents an average of five measurements. The average scatter is of the order of  $\frac{1}{2}$  7%.

- ISML tests

Each point represents the average of two tests. The scatter is much smaller.

- 74 -

- Comparison of the two results:

The values obtained by Euratom are higher by about 4), than those of ISML over the whole range of  $Al_00_3$  contents.

#### Dynamic method:

As foreseen, the dynamic Young's modulus is higher than the static Young's modulus (about 12%).

# 3.5.1.2.2 <u>Variations in Young's modulus with the temperature</u> for four SAP ISML contents.

#### Static method:

For the SAP 4%, the Euratom results show a discontinuity between 250 and  $350^{\circ}$ C. The lack of agreement with the ISML results is of the order of 3%.

#### Dynamic method:

The dynamic Young's modulus was only determined on SAP 4%, and is about 20% higher than the static modulus.

#### 3.5.2 Linear Thermal Expansion

#### 3.5.2.1 Test conditions

- <u>Apparatus</u> : A Chevenard-type dilatometer with photographic recording was used. It was a special Adamel Type 55 equipped to operate under vacuum or in controlled atmosphere.
- Test specimen : A 50 cm long specimen was used for measuring the expansion in the extruded direction and a 25 cm specimen for the perpendicular direction.
- Experiment : This was conducted in argon atmosphere with an increasing temperature of 60°C/hr. The magnification K was 150 or 300 times.

#### 3.5.2.2 Experimental results

The results obtained perpendicular or parallel to the extrusion direction were practically identical. The results for the latter direction are given in Fig. 94. The values for the linear thermal expansion are plotted as a function of the oxide content for several temperature ranges between 20 and 500<sup>0</sup>C.

It was possible to establish the equation below expressing the length of SAP samples of different grades as a function of the temperature:

SAP ISML 4%  $l_t = l_0 (1 + 21.32 \cdot 10^{-6}t + 9.10^{-9}t^2)$ SAP ISML 7%  $l_t = l_0 (1 + 20.38 \cdot 10^{-6}t + 9.10^{-9}t^2)$ SAP ISML 10%  $l_t = l_0 (1 + 19.73 \cdot 10^{-6}t + 9.10^{-9}t^2)$ SAP ISML 14%  $l_t = l_0 (1 + 18.93 \cdot 10^{-6}t + 9.10^{-9}t^2)$ 

It can again be seen from these formulae that the thermal expansion drops as the oxide content increases.

#### 3.5.3 <u>Electrical Resistivity</u>

#### 3.5.3.1 Test conditions

Euratom laboratory (Ref. 50)

The tests described were performed with a double Thompson bridge. The electrical resistance of the bridges was verified to an accuracy of better than 2  $\cdot$  10<sup>-4</sup>. The test specimen is a cylinder 6 mm in diameter and 80 mm long. The sample holder was designed specially in order to increase the relative precision of the gauge length up to 0.1%. Under the above conditions, the relative error in the determination of the resistivity ( $\Delta R$ ) is less than 0.5%.

Euratom laboratory (Ref. 54)

The electrical resistivity was measured by the d.c. potentiometric method. The samples were mounted in series with a standard resistance, immersed in a thermostatic bath and the current flow then measured with a potentiometer. The limit accuracy in the determination of the current was that of the standard, i.e.,  $2 \cdot 10^{-4}$ . The samples were immersed in a thermostatic bath kept at 25 ± 0.05°C and the potential drop between two knife edges measured with a five dial potentiometer. Care was taken to eliminate thermoelectric emf by reversing the current.

The samples were cylinders 4 mm in diameter and 50 mm long. The error affecting the dimensions can be estimated at <1%. All the samples had been annealed for two hours at 550°C in a high-vacuum furnace.

The resistivity at high temperature up to  $500^{\circ}$ C was measured by determining the ratio between the resistances at the given temperature and at 25°C. The resistivity data at high temperature were corrected for the effect of thermal expansion.

As a coefficient of thermal expansion of SAP, a value of  $24.10^{-6}$  cm/<sup>o</sup>C was taken which is independent of the orientation (Ref. 25).

The samples were heated in a high-vacuum furnace stabilized to  $\pm 0.5^{\circ}$ C, standardized thermocouples being used. The reproducibility of the electrical resisticity data was to within  $2.10^{-3}$  and it can be assumed that the error affecting these values is  $\leq 1\%$ .

ISML Laboratory (Ref. 9).

The classical d.c. potentiometric method was used, the testing specimen being connected in series with a calibrated resistance. The testing specimen is machined from an extruded plate ( 30 x 2.5 mm) and annealed for 24 hr at 500°C. The tests were conducted up to 550° in a furnace with a temperature determination of  $\pm$  1°C. The accuracy of the readings of the potentiometer was 10<sup>-7</sup>. The relative scatter in the results observed in several tests in the same conditions did not exceed 3.10<sup>-4</sup>. The ratio  $\frac{R_t}{R_{20}}$  was determined for every temperature and the absolute value of  $R_{20}$  for every percentage of  $Al_2O_3$ .

3.5.3.2 <u>Results</u>

# - <u>Resistivity as a function of the oxide content at 20<sup>°</sup>C</u> (Ref. 50, Euratom, Fig. 95)

On this diagram we have also plotted the values determined by other laboratories. This diagram clearly shows that the absolute resistivity increases with the oxide content. Every test specimen was chemically analyzed, and there is a relatively high scatter in the oxide content.

The mean scatter of the resistivity is in the range of  $\pm$  3.5%. The values obtained in other laboratories for other batches are in the normal scatter range.

- <u>Resistivity as a function of the test temperature for</u> <u>different grades of SAP ISLL</u> (Refs. 9, 26 and 54, Figs. 96 and 97, Euratom and ISML results)

The electrical resistivity increased with temperature and the results obtained in the two laboratories are in good agreement.

- Resistivity parallel and perpendicular to the direction of extrusion as a function of the oxide content at  $100^{\circ}C$  and  $500^{\circ}C$ 

Euratom result (Ref. 54, Fig. 98)

The resistivity values increase with the oxide content, as seen above (Fig. 95). Moreover, the transverse resistivity is higher than the longitudinal one.

#### 3.5.4 Thermal conductivity

#### 3.5.4.1 Experimental procedure

## Euratom laboratory (Ref. 53)

The apparatus is Euratom-built. The method is a stationary one, conducted with axial flow along a cylindrical sample. Armco iron was selected as the reference sample.

#### Euratom laboratory (Ref. 54)

For this present study, use was made of a new transient flow method developed to meet the specific requirements of our research. This method enables both the thermal diffusivity and the conductivity in a given direction to be determined and offers the advantage of using a known thermal capacity as a reference. Because the specific heat is a property known with much greater accuracy than the thermal conductivity, the method seems more reliable than those based on comparison with a standard thermal resistance.

Comparative determination on high purity nickel and aluminium samples with conventional steady state techniques and the methods described above resulted in close agreement, the difference being in all cases less than 6%. The relative method employed by the US Bureau of Standards (Ref. 44) for use up to  $600^{\circ}$ C was adopted. The method consists in measuring the thermal gradient along two cylindrical samples, one of them being the material to be tested and the other one a reference sample (in this case lead). The two samples are welded together at the end and placed in a metallic tube. A complete description of this apparatus is given in Ref. 66.

## 3.5.4.2 <u>Results</u>

- <u>Thermal conductivity plotted as a function of the temperature</u> (Figs. 99, 100 and 101).

The ISML laboratory expressed the thermal conductivity of SAP ISML as a proportion of that of aluminium (Raffinal 99.995%).

The apparatus used by the Euraton laboratory (Ref. 53) was tested on 99.999% aluminium and the values obtained plotted in Fig. 99 are in good agreement with the literature.

From Figs. 99, 101, it can be seen that the conductivity falls compared with that of aluminium as the oxide content increases.

In conclusion, it may be said that the conductivity seems to drop as the temperature rises but the results obtained by the three laboratories do not seem to be in good agreement.

- <u>Thermal conductivity</u>, <u>longitudinal and transverse at 100<sup>°</sup>C and</u> 500<sup>°</sup>C versus the oxide content (Ref. 54, Figs. 102 and 103)

The transverse conductivity is always lower than the longitudinal one. The difference increases as the oxide content rises, but is fairly constant for the temperature related.

- Thermal conductivity, longitudinal and transverse, of SAP ISML 10% versus the temperature (Ref. 53, Fig. 99)

The transverse conductivity is 25% lower than the longitudinal one and this difference decreases slightly as the temperature rises.

## 3.5.5 Density

The values for the density versus the percentage of  $Al_2O_3$  are plotted in Fig. 104.

These results concern batches of SAP ISML which are representative of the production from 1960 to 1966. The scatter of the results does not exceed  $\pm$  0.2%. It can be seen that the density is proportional to the percentage of Al<sub>2</sub>0<sub>3</sub>. Fig. 104a shows results obtained at Risö Institute (Ref. 41). The density of the materials was determined according to Ref. 39. The data are averages of 3-6 measurements.

#### - 81 -

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