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EUROPEAN ATOMIC ENERGY COMMUNITY - EURATOM

IMPROVEMENTS IN SAP TECHNOLOGY  
WITH A VIEW TO NUCLEAR APPLICATIONS  
PROPERTIES OF THE MATERIALS STUDIED

by

D. GUALANDI (I.S.M.L.) and P. JEHENSON (EURATOM)

1963



ORGEL Program

Report established under the contracts EURATOM/MONTECATINI  
No 003-60-5 ORG I and No 026-61-7 ORG I

Text presented at the «Journées Métallurgiques d'Automne»  
Paris, 15-19 October 1962

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— SAP 960	at about 4 % $Al_2O_3$
— SAP 930	at about 7 % $Al_2O_3$
— SAP 895	at about 10 - 11 % $Al_2O_3$
— SAP 865	at about 13 - 14 % $Al_2O_3$

The principal improvement on the semi-finished products was obtained by a degassing thermal treatment under vacuum (about twenty hours at 600-625° C, under  $10^{-4}$  —  $10^{-5}$  mm. Hg) of the powders after cold compaction. The main consequences of this special treatment on SAP, then named SAP-ISML, are the following ones :

- 1) an important decrease of the residual gas content, which becomes lower than 10 ppm ;
- 2) consequently, an excellent high temperature (600° C) stability and a considerable improvement of the welding properties.

The paper also gives some important characteristics of the four SAP-ISML grades, mechanical properties at room and at high temperature, fatigue and creep resistance, pressure tests and hot stability. It also gives the actual tolerances obtained in the fabrication of fuel element and pressure tubes.

A very large dispersion of the results must probably be attributed to the non homogeneity of the powders. However, comparison of the actual results allows to select SAP-ISML 930 for the fabrication of fuel element tubes and SAP-ISML 895 for the fabrication of pressure tubes ; this SAP-ISML 895 has to be considered as a limit grade for the fabrication of fuel elements tubes.

It is planned to extend this work in order to improve the quality of the initial powders (homogeneity, purity) and the fabrication process in itself.

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## C O N T E N T S

1. GENERAL REMARKS.
2. IMPROVEMENTS IN SAP TECHNOLOGY.
  - 2.1. General manufacture chart.
  - 2.2. Improvements in SAP Technology.
3. PROPERTIES OF SAP PRODUCTS.
  - 3.1. Thermal stability.
  - 3.2. Manufacture.
  - 3.3. Mechanical properties.
4. ACTUAL CONCLUSIONS ON SAP-ISML.
  - 4.1. Quality.
  - 4.2. Nuclear applications.
    - 4.2.1. SAP-ISML 960 and 930
    - 4.2.2. SAP-ISML 895 and 865.
  - 4.3. Actual work.





IMPROVEMENTS IN SAP TECHNOLOGY  
WITH A VIEW TO NUCLEAR APPLICATIONS  
PROPERTIES OF THE MATERIALS STUDIED

SUMMARY

This paper describes some experimental work done under EURATOM contract at I.S.M.L. (Istituto Sperimentale Metalli Leggeri, Novara, Italy), in close cooperation with the Metallurgy and Ceramics Service of the EURATOM Common Research Center of Ispra ; this experimental work represents the initial stage of a long range research program on SAP, in the field of the ORGEL project.

The SAP powders used in this research are commercial powders produced by the Swiss Company A.I.A.G. ; this company owns the SAP patents. The grades are :

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The principal improvement on the semi-finished products was obtained by a thermal degassing of the powders after cold compaction (about twenty hours at 1100 - 1150° F, under  $10^{-4}$  -  $10^{-5}$  mm.Hg). The main consequences of this special treatment on SAP, then named SAP-ISML, are the following ones :

- 1) an important decrease of the residual gas content, which becomes lower than 10 ppm ;
- 2) consequently, an excellent high temperature (1100° F) stability and a considerable improvement of the welding properties.

The paper also gives some important characteristics of the four SAP-ISML grades (mechanical properties at room and at high temperature, fatigue and creep resistance, pressure tests and thermal stability).

A very large dispersion of the results must probably be attributed to the non homogeneity of the powders. However, comparison of the actual results allows to select SAP-ISML 930 for the manufacture of fuel element sheaths and SAP-ISML 895 for the manufacture of pressure tubes ; this SAP-ISML 895 has to be considered as a limit grade for the manufacture of fuel elements sheaths.

It is planned to extend this work in order to improve the quality of the initial powders (homogeneity, purity) and the manufacture in itself.

## 1. GENERAL REMARKS.

There are some nuclear power reactors in which aluminum could be used as far as corrosion resistance is concerned and because of its low neutron absorption cross section but the possibilities of using this metal are hindered by the low mechanical strength at high temperature, for instance 550°F to 900° 1.

The use of conventional aluminum alloys, hardened by proper aging treatment or cold-work, does not overcome this difficulty because at the above mentioned temperatures aluminum alloys hardened by precipitation or cold-work lose their properties.

A better prospect is given by a material obtained with powder metallurgy, a sintered aluminum product called SAP.

EURATOM in the field of the ORGEL project undertook in 1960 some work on SAP ; this work was done under contract at I.S.M.L. (Istituto Sperimentale Metalli Leggeri, Novara, Italy) in close cooperation with the Metallurgy and Ceramics Service of the EURATOM Common Research Center of Ispra.

To get SAP, a special aluminum powder is used (fig. 1a and 1b) in which, with a hammering and mixing process in a ball-mill, the aluminum is partly transformed into  $Al_2O_3$  - alumina - and the particles of alumina are embedded into the aluminum lamellae .

After compacting and sintering of the powder, the material (SAP) has a structure as shown in fig. 2 : it is an aluminum matrix in which very fine  $Al_2O_3$  particles are dispersed.

This structure, typical of a "dispersion" hardened material is similar to the structure of an age hardened alloy (fig. 3) with the important difference that the dispersed phase is not soluble in aluminum even at temperatures as high as the aluminum melting point; therefore the material can better maintain at high temperature good mechanical properties.

SAP was patented by the Swiss Company AIAG, and in our study, we used powders produced by this company, and having 4 different contents of  $Al_2O_3$  :

- SAP 960                      average 4 %  $Al_2O_3$
- SAP 930                      average 7 %  $Al_2O_3$
- SAP 895                      average 10 % - 11 %  $Al_2O_3$
- SAP 865                      average 13 % - 14 %  $Al_2O_3$ .

On the typical properties of commercial SAP and on its preparation modalities, we give reference to a good summary of the knowledge on the SAP production (1).

The purpose of our research was to improve the manufacture process in order to obtain a material more suitable for use in nuclear plant because commercial products were bringing difficulties owing to their tendency to give blisters when annealed for some time at sufficiently high temperatures.

In the field of the ORGEL project, the fuel element sheath must stand temperatures in the range of  $750^\circ F - 850^\circ F$  ; the coolant is an organic compound (terphenyls) and the moderator, heavy water.

Aluminum is foreseen for fuel element sheaths because of its low neutron absorption cross-section in comparison with stainless steel and of its good corrosion resistance towards the terphenyls as compared with other materials like zirconium alloys.

The typical case in which  $Al_2O_3$  dispersion in aluminum can be used is defined by the following requirements :

- 1) sufficient mechanical strength at the foreseen temperatures ( $750^\circ - 850^\circ F$ ) ;
- 2) stability of the mechanical properties, also under stress (creep-resistance) ;

- 3) possibility of making finished products with the close dimensional tolerances required ; finned tubes, tubes with thin walls, etc. ;
- 4) good structural stability at the temperatures foreseen (no blisters, no microcracks) ;
- 5) sufficient weldability, with absolute tightness of the welded joints in order to avoid leaks of fission products ;
- 6) good compatibility with the organic coolant and with the nuclear fuel (Uranium oxide or carbide) ;
- 7) mechanical properties and general behaviour should not change also during irradiation.

## 2. IMPROVEMENTS IN SAP TECHNOLOGY.

### 2.1. General manufacture chart :

Our research was then mainly aimed to produce tubes for fuel element sheaths that could meet the above mentioned specifications, with diameters in the order of  $1/2$  of an inch to 1 inch and wall thickness from  $1/50$  th of an inch to  $1/25$  th of an inch.

The general method for making tubes is described in the literature (1) ; we will only summarize the general chart :

- Preparation of the powder with the nominal  $Al_2O_3$  content (4, 7, 10 or 14 %) ;
- Cold compacting of the powder ( $\sim$  14 tons/in.<sup>2</sup>) ;
- Heat treatment at 1000° F and hot compacting ( $\sim$  35 tons/in.<sup>2</sup>) ;
- Extrusion and cold drawing of the tubes

### 2.2. Improvements in SAP technology :

As we said before, a commercial product made by this method is not interesting for the nuclear applications foreseen because it has a tendency to give blisters or to develop internal flaws.

The mechanism of formation of these defects is complex under some respects but is certainly related to the fact that commercial products contained remarkable quantities of gases, mainly hydrogen, in the order of 50 to 100 parts per million ; that corresponds roughly to 50 to 100

cubic centimeters of  $H_2$  (at room temperature) for every 100 grams of material. This can easily explain the blisters, the internal microcracks and also the difficulties to confer good welding properties.

We thought that the best way to get a good material from this point of view was to submit the product to a degassing treatment in a vacuum furnace at a proper temperature.

There were three possibilities :

- degassing treatment of the powders ;
- degassing treatment during manufacture ;
- degassing treatment of the finished products.

All these three possibilities were checked and the conclusion of our work was that it is better, both from the efficiency as well as from the economical point of view to have the degassing treatment during the manufacture (11).

There is also a good reason for this choice : we said before that after cold compacting the specimens are heated before the hot compaction.

During this thermal treatment, the amorphous alumina present in the powders is transformed into more stable cristalline  $Al_2O_3$ . To get a complete transformation it is necessary to leave the material for some time at a sufficiently high temperature ( $1000^\circ F$  for at least 15 hours). Now, this thermal treatment is conducted on cold compacted material in a vacuum furnace ( $10^{-4}$  -  $10^{-5}$  millimeters mercury) during about 20 hours at  $1100-1150^\circ F$ , in order to have at the same time, alumina stabilization and hydrogen elimination.

In fig. 4 we show the correlation found between the final hydrogen content and the treatment conditions (temperature, time,  $Al_2O_3$  content).

It is clear that the lower the  $Al_2O_3$  content the better the degassing.

It is important to have temperatures comprised between  $1100^\circ F$  and  $1200^\circ F$ , the time of treatment being of about 20 hours. The limits attained in gas content are 1 - 2 parts per

million with 4 %  $Al_2O_3$  to 6 - 8 parts per million with 14 %  $Al_2O_3$ .

For this type of modified treatment to produce SAP, a patent application was made in may 1961, and a paper was presented at the "Giornate dell' Energia Nucleare" Milan - december 1961 (11).

### 3. PROPERTIES OF SAP PRODUCTS :

#### 3.1. Hot stability :

With this material, called SAP-ISML to differentiate it from commercial SAP, we never observed blisters and even microcracks were only found very rarely in specimens heated to temperature approaching the aluminum melting point.

In fig. 5, there is a comparison between some extruded shapes of commercial SAP and similar shapes of SAP-ISML treated several hours at 1100°F ; that shows the good behaviour of the vacuum treated material.

#### 3.2. Manufacture :

Some typical products obtained during this work are shown in figs. 6 and 7 : they are finned tubes twisted after extrusion or obtained with helical fins by direct extrusion with a special extrusion die.

It was also developed a method of welding SAP plugs by hot pressure on SAP tubes : some dummy elements obtained with this method, also patented, are shown in fig. 8.

Some bursting tests conducted at room temperature or at 900°F on those welded plugs are shown in fig. 9 : the burst occurred outside the welded zone.

#### 3.3. Mechanical properties :

We will now briefly give an outline of the general mechanical properties of our material.

Fig. 10 shows the mechanical properties of extruded tubes (of two different diameters) vs the oxide content.

In the following three figs. 11, 12, 13, the average values of the ultimate tensile strength, the yield limit and the elongation are given (vs temperature) for the 4 different qualities of SAP-ISML. These values are average ones : there is a rather large dispersion in the mechanical properties, that we are now trying to minimize ; actually we think that this large dispersion is probably due to the non homogeneity of the powders.

Impact resistance versus temperature is given in fig. 14.

If SAP-sheet are subjected to cold-work, mechanical properties at room temperature change as it is shown for the quality with 7 % alumina in fig. 15 : an annealing treatment (2 hours at 930° F) partly recovers the original properties as it is shown in the same figure (dotted lines).

As far as fatigue is concerned we show in fig. 16 the fatigue curves for the 4 types at room temperature, taken on specimen without any notch. With a U notch, the corresponding curves are shown in fig. 17. It is interesting to note that a different type of notch (V type) does not lower the curve more than a U type notch with the same depth ; in figs. 18 and 19, this is shown for the 4 % and 7 %  $Al_2O_3$  grades. We have also conducted some fatigue tests at 750° F ; the curve is shown (for the 4 %  $Al_2O_3$ ) in fig. 20.

Just one more word about creep resistance : in figure 21 is shown the life vs stress curves for the 4 % and 7 %  $Al_2O_3$  grades and in fig. 22 the similar curves for the 10 % and 14 % grades. The creep resistance is the longitudinal one ; we have also tested tubes under different pressures of nitrogen, in order to have their life before burst vs pressure. In fig. 23 the results are shown for the 4 % and 7 %  $Al_2O_3$  grades. There is a fairly large dispersion of the results ; nevertheless we can say that the lowering of creep resistance in tubes (stressed in the transverse direction) is about 25-30 % the longitudinal creep resistance.

4. ACTUAL CONCLUSIONS ON SAP-ISML :

4.1. Quality :

The vacuum treatment improves considerably the properties of sintered aluminum products, particularly the stability at high temperature and consequently the weldability. This depends upon the lowering of the hydrogen content in the finished products, to 10 ppm or less ; consequently blisters and internal flaws are suppressed.

4.2. Nuclear applications :

In the field of the ORGEL project, we can give the actual following conclusions :

4.2.1. SAP-ISML 930 and 960 :

The manufacture difficulties are the same with SAP-ISML 960 and SAP-ISML 930 ; the high temperature properties of SAP-ISML 930 are better than those of SAP-ISML 960 (mechanical properties, creep resistance, burst tests) ; consequently, it seems that SAP-ISML 930 is more interesting than SAP-ISML 960 for the manufacture of fuel element sheaths.

4.2.2. SAP-ISML 895 and 865 :

SAP-ISML 895 and 865 grades were initially considered for the manufacture of pressure tubes. Although SAP-ISML 865 has better high temperature properties, manufacture of good quality tubes of 90 mm internal diameter and of about 2 mm thickness is very difficult in SAP-ISML 865 ; consequently we now prefer SAP-ISML 895, which can also be considered as a limit grade for the manufacture of fuel element sheaths.



4.3. Actual work :

This work is still going on to improve the quality of the initial powders (homogeneity, purity) and the manufacture in itself.

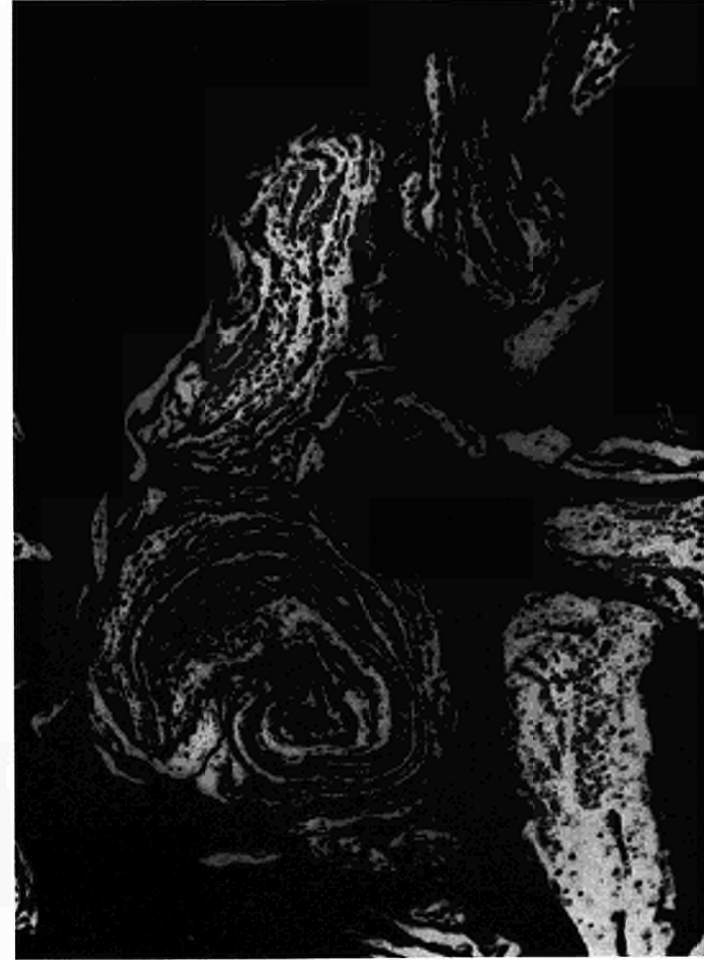
Acknowledgements :

The authors wish to acknowledge the contribution of the ISML and EURATOM collaborators who have taken part in the work described in this paper.





a)



b)

FIG. 1

Microstructure of SAP-type powders  
magnification : a) X 500 - Etch : HF 0,5 %  
b) X 1000

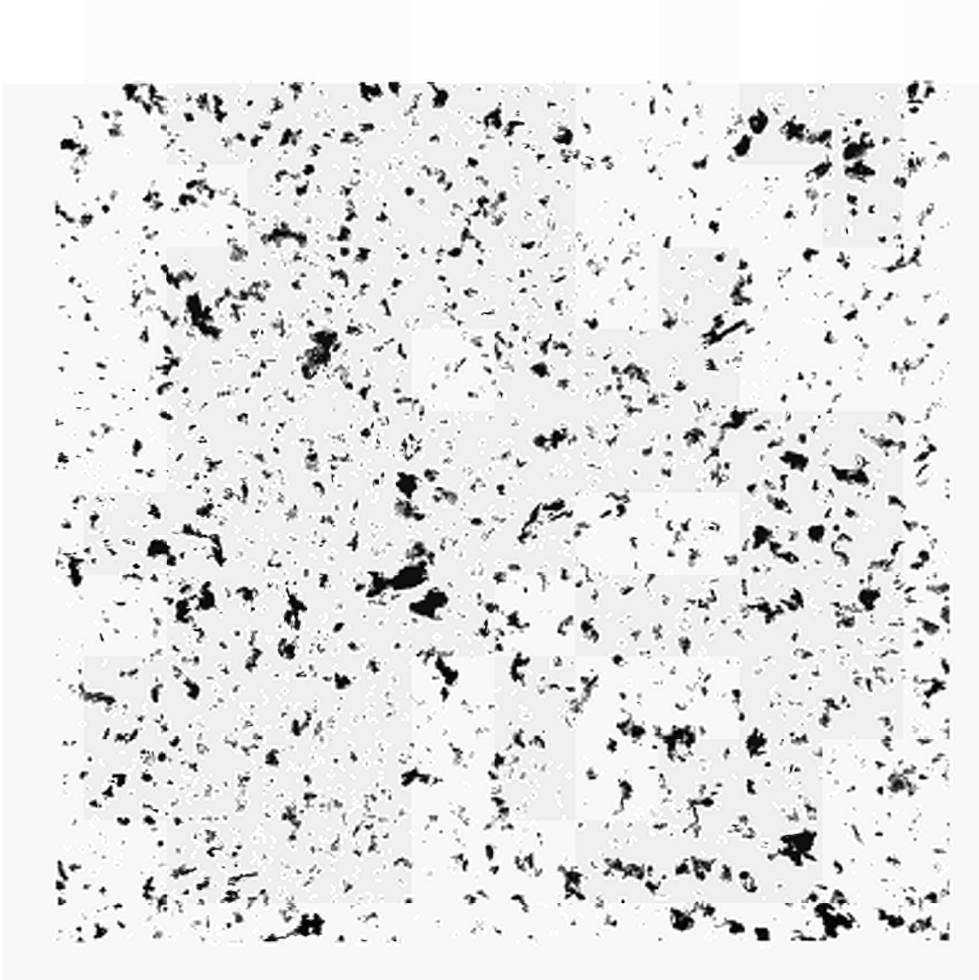
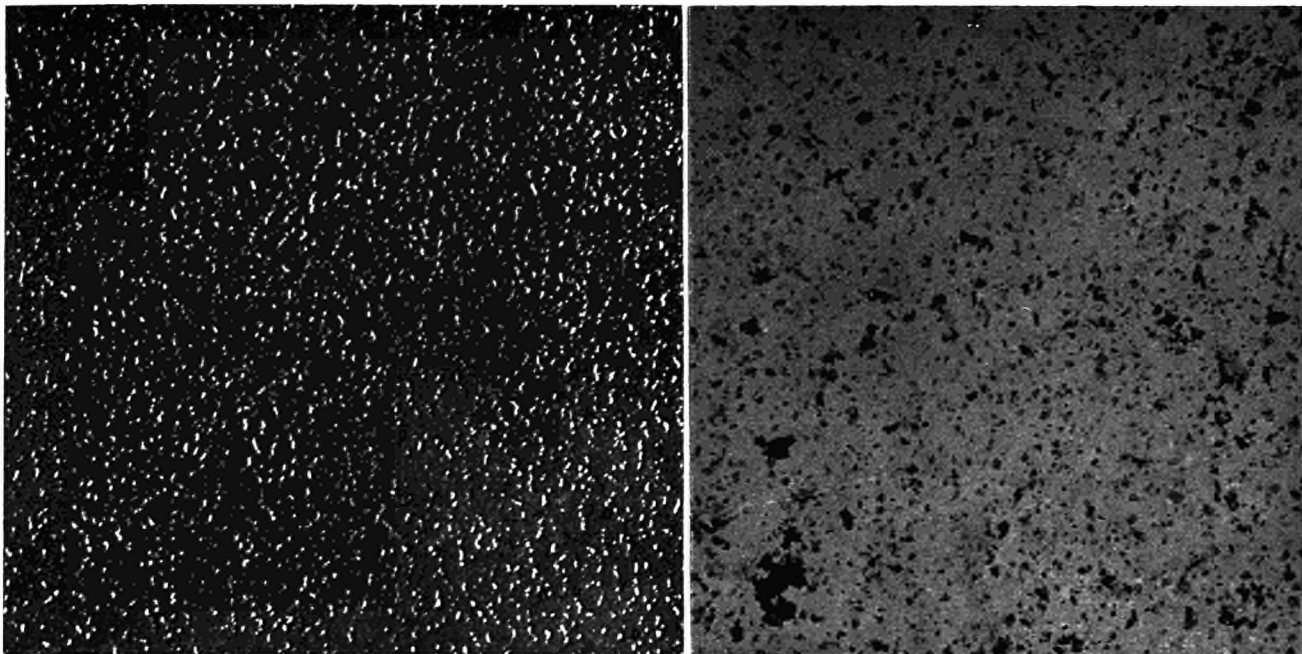


FIG. 2

Structure of SAP-ISML 7 % Al<sub>2</sub>O<sub>3</sub> at the electron microscope.  
Original magnification : X 20.000



Al - 5 % Cu

SAP-ISML 7 %

FIG. 3

« Precipitation » hardened-alloy structure vs.  
« Dispersion » hardened structure of SAP-ISML 7 %  $Al_2O_3$ .

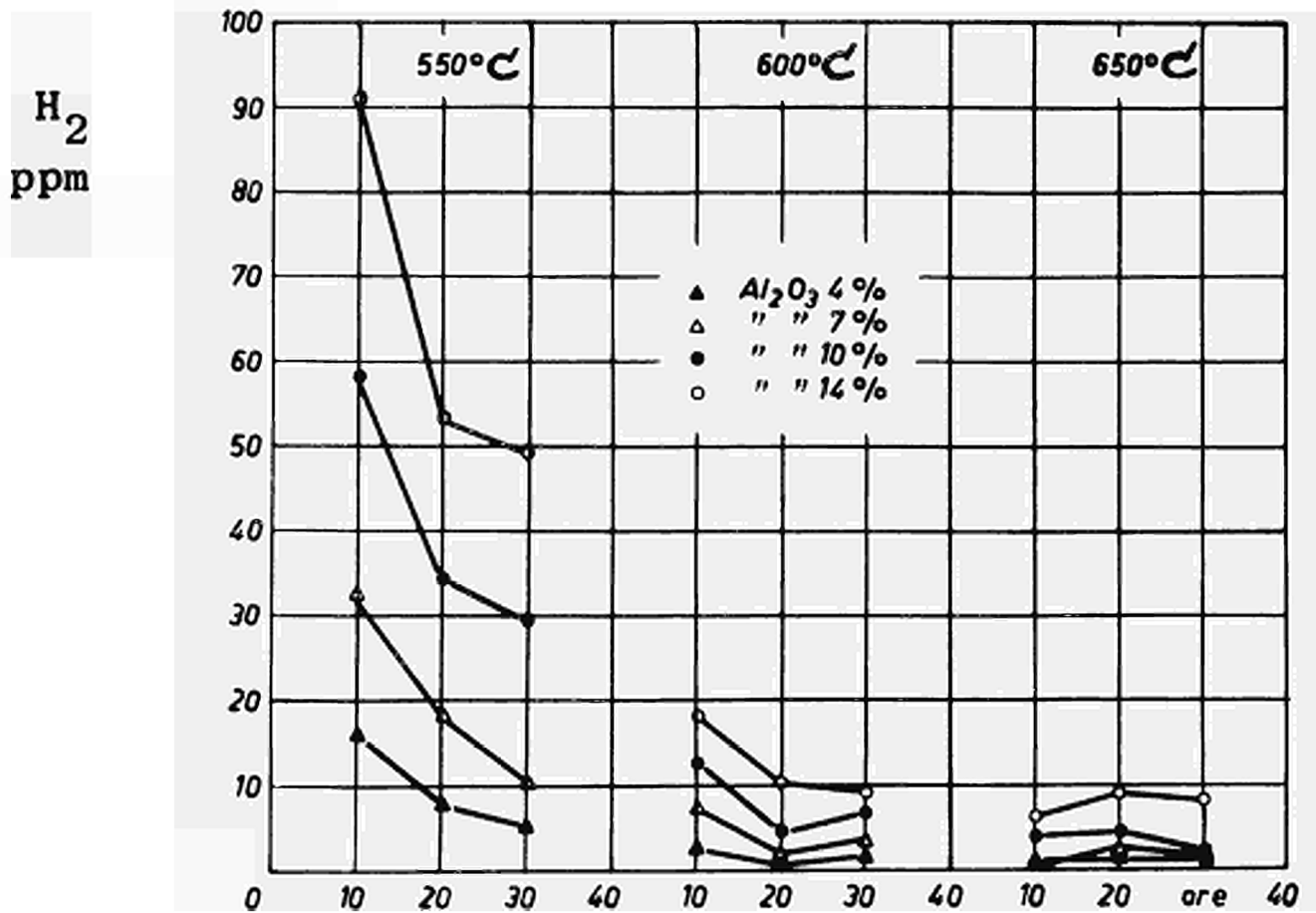


FIG. 4

Hydrogen content of SAP products depending on temperature of heat treatment and time of treatment.

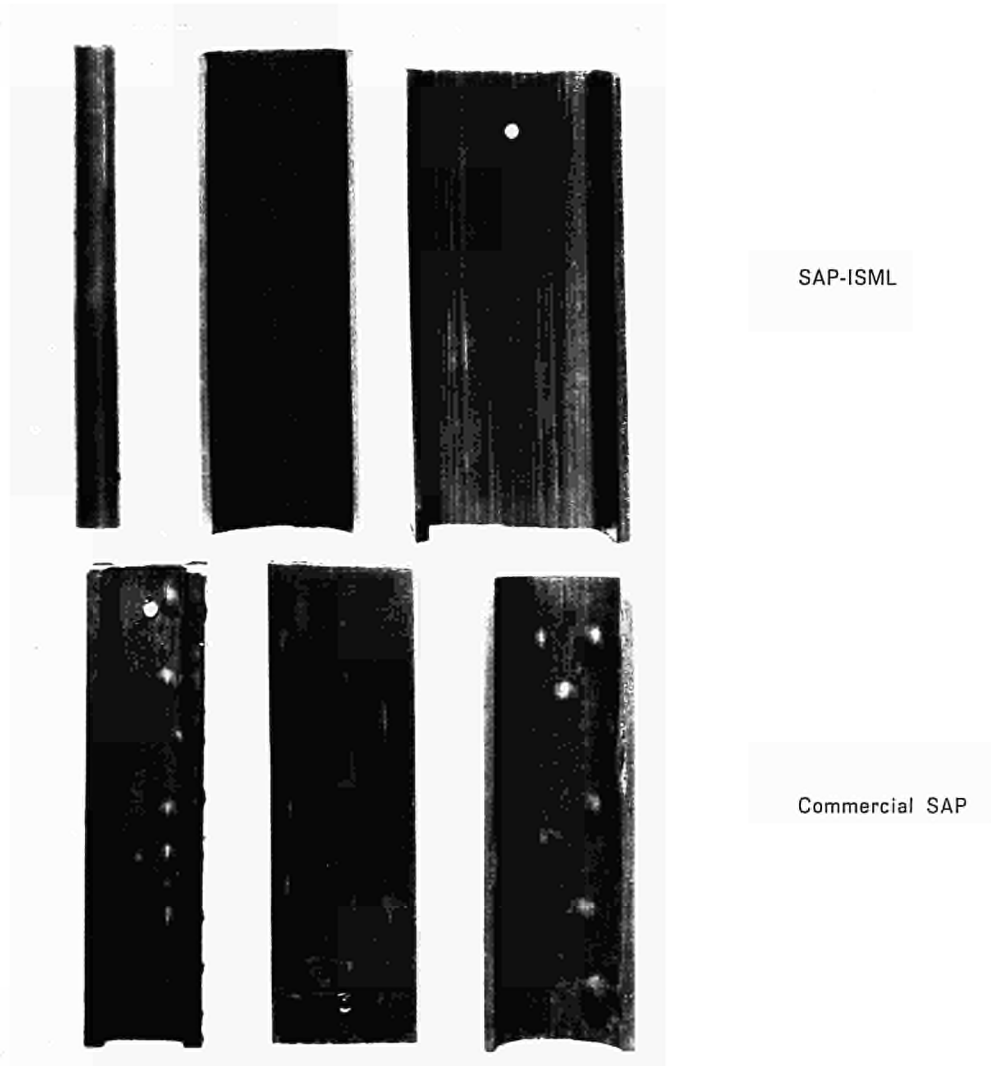


FIG. 5

Different behaviour of commercial SAP when heated in air (blisters) vs. SAP-ISML treated under vacuum during fabrication.

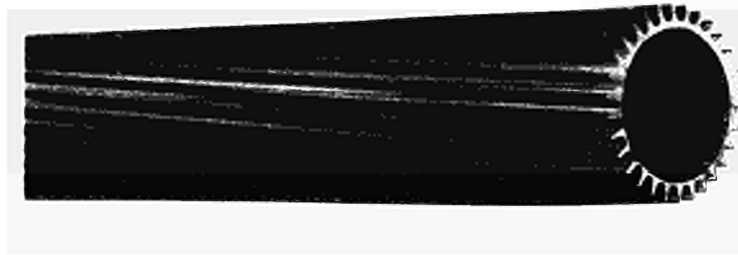


FIG. 6

Tube of SAP-ISML 7 %  $Al_2O_3$  with 30 fins twisted after the extrusion.



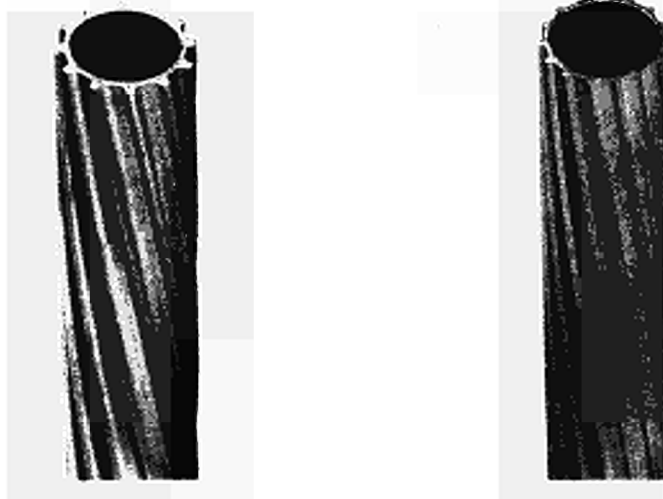


FIG. 7

Tubes of SAP-ISML 7 % Al<sub>2</sub>O<sub>3</sub> with 12 and 15 fins obtained directly during extrusion.

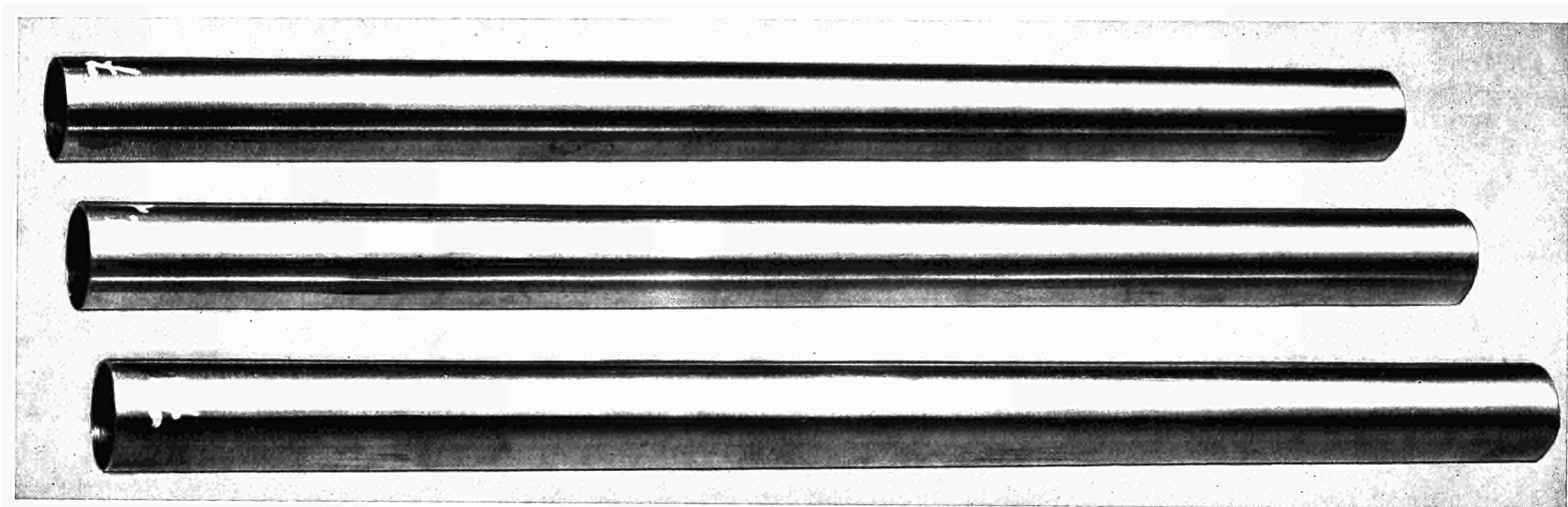


FIG. 8

Dummy elements with SAP-ISML 7%  $\text{Al}_2\text{O}_3$  sheaths.



FIG. 9

Tube of SAP-ISML 4 % Al<sub>2</sub>O<sub>3</sub> closed at one end by hot-pressure method after bursting test at room temperature and at 900° F.

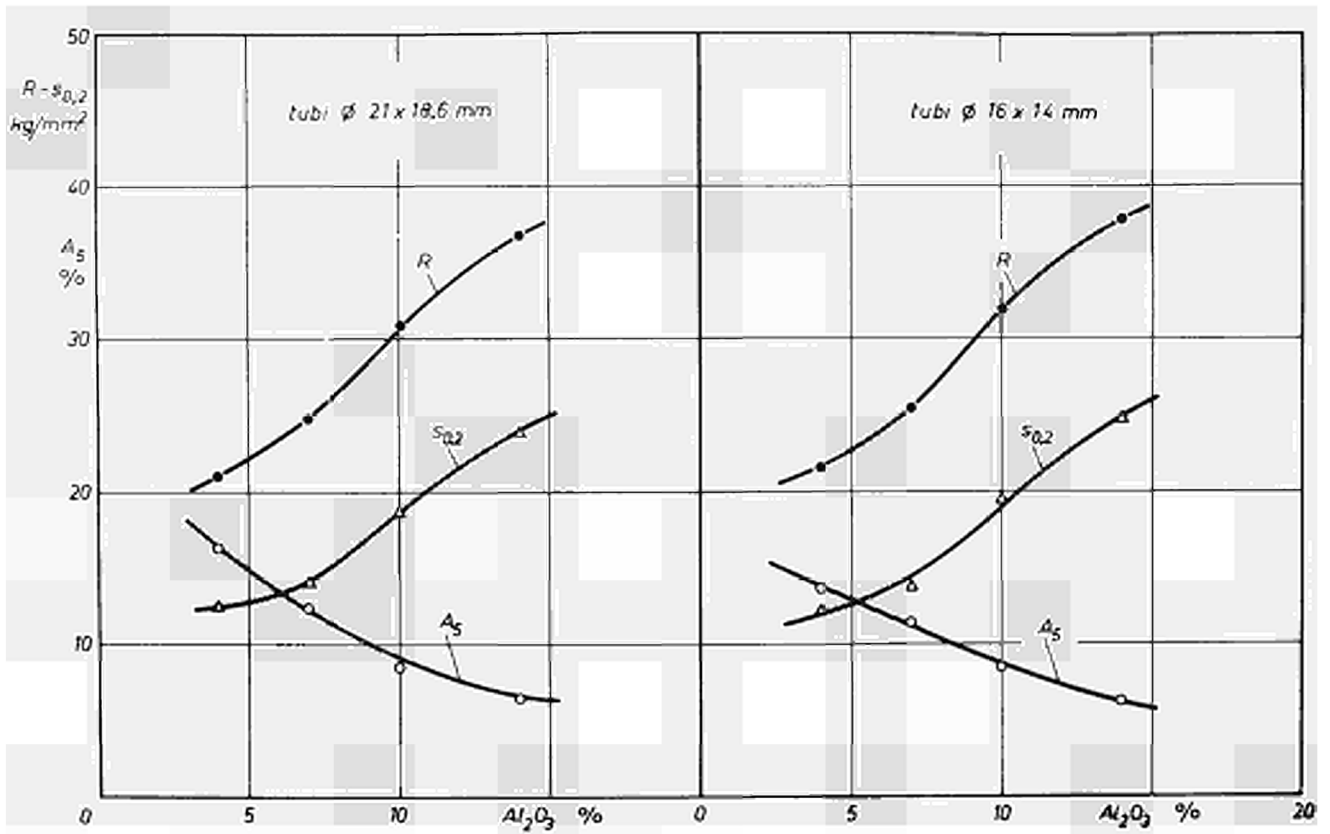


FIG. 10

Mechanical properties of extruded tubes of SAP-ISML vs. content of  $\text{Al}_2\text{O}_3$ .

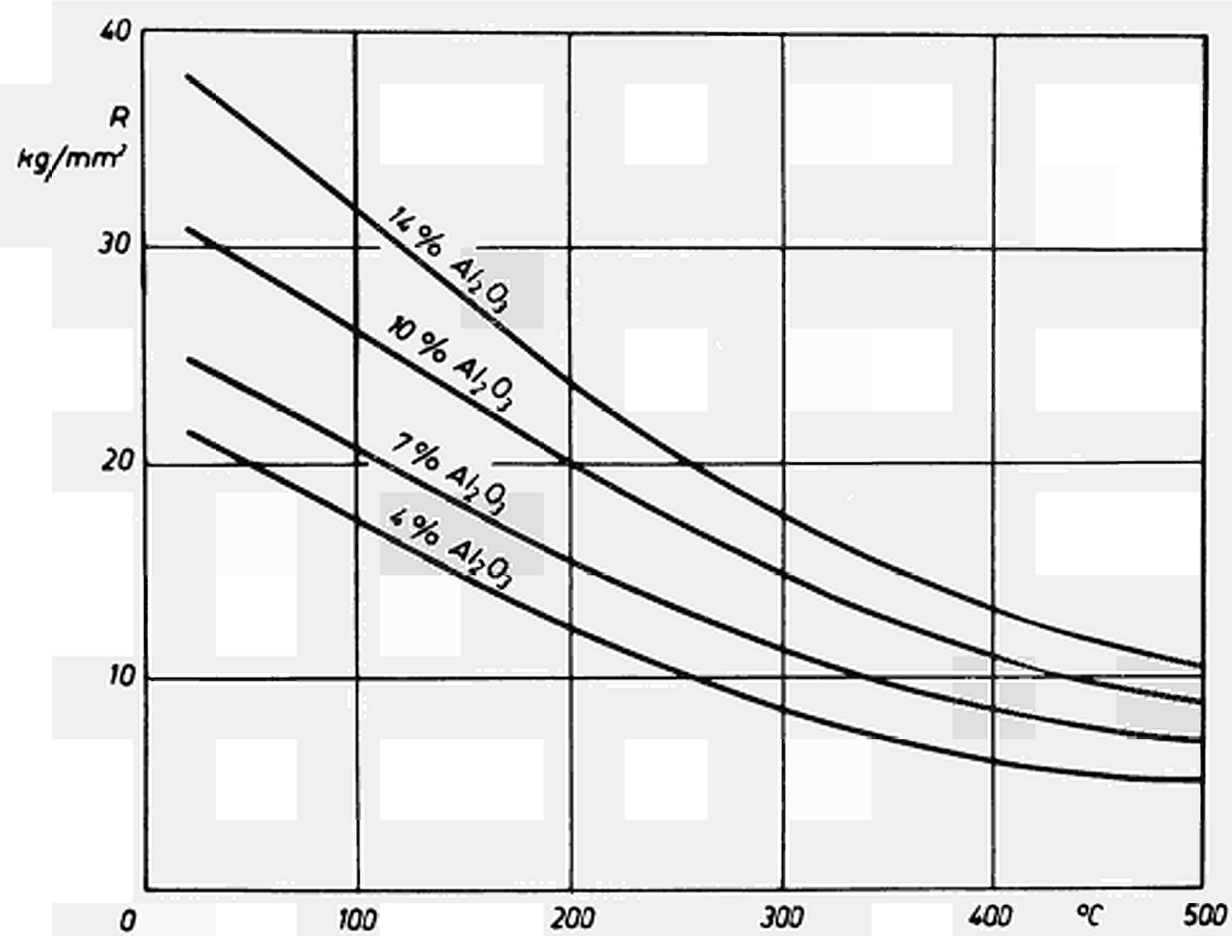


FIG. 11

Average values of rupture strength of SAP-ISML (at different levels of  $Al_2O_3$ ) vs. temperature.

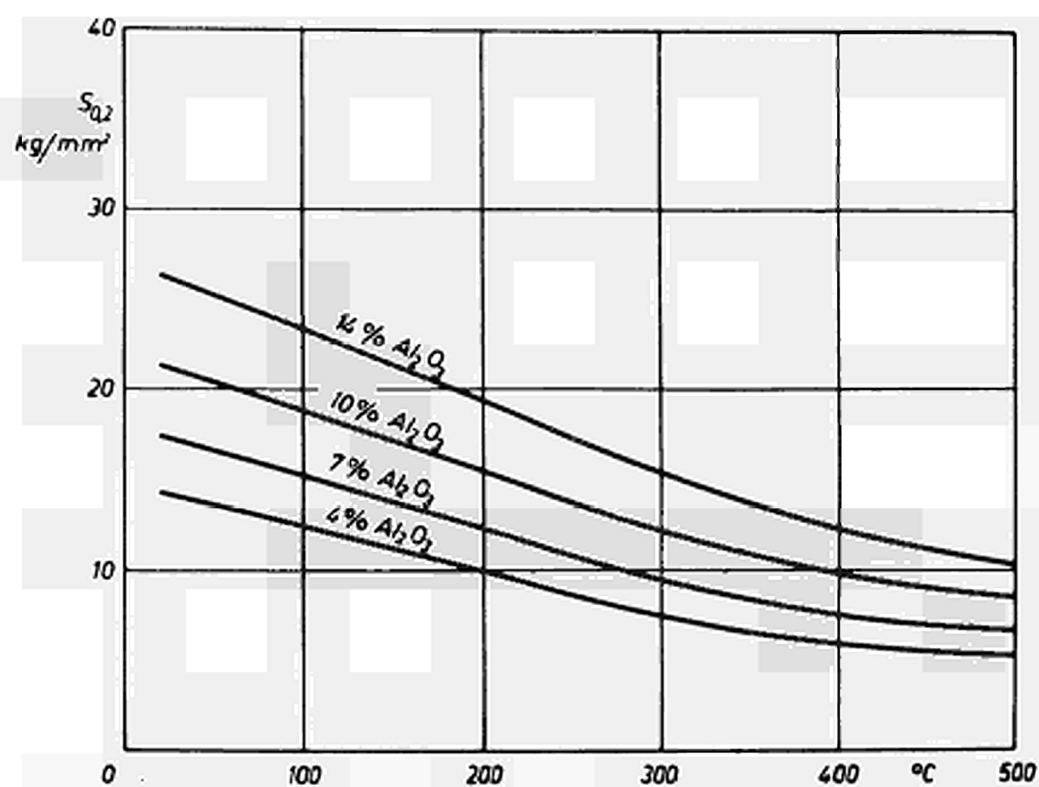


FIG. 12

Average values of yield limit of SAP-ISML (at different levels of  $\text{Al}_2\text{O}_3$ ) vs. temperature.

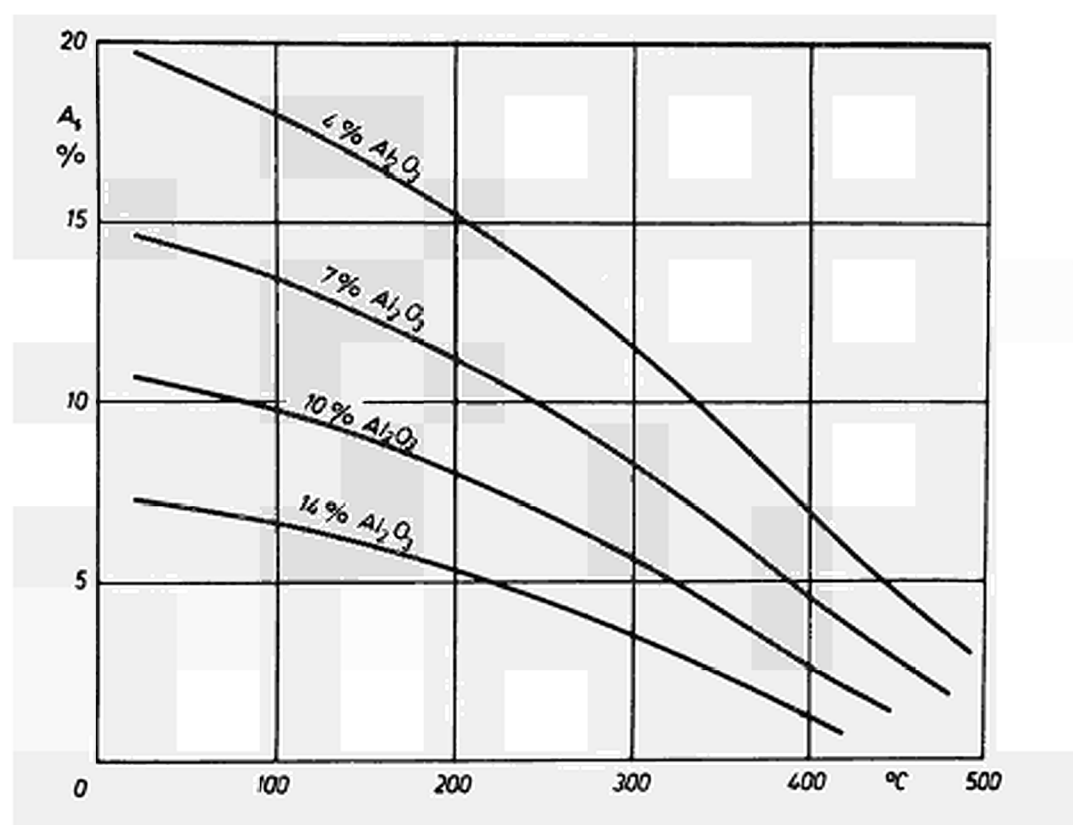


FIG. 13

Average values of elongation of SAP-ISML (at different levels of Al<sub>2</sub>O<sub>3</sub>) vs. temperature.

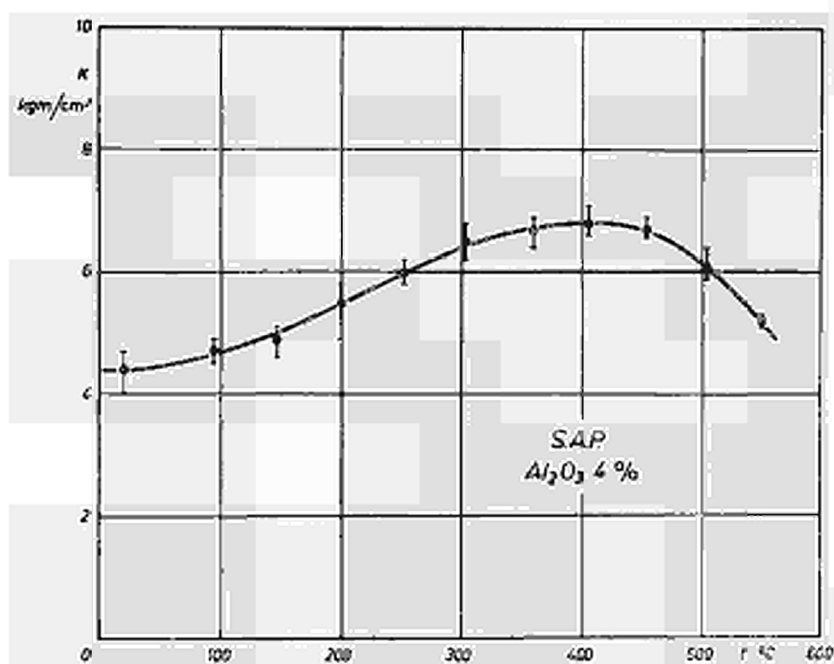


FIG. 14

Impact resistance of SAP-ISML 4 %  $\text{Al}_2\text{O}_3$  vs. temperature.



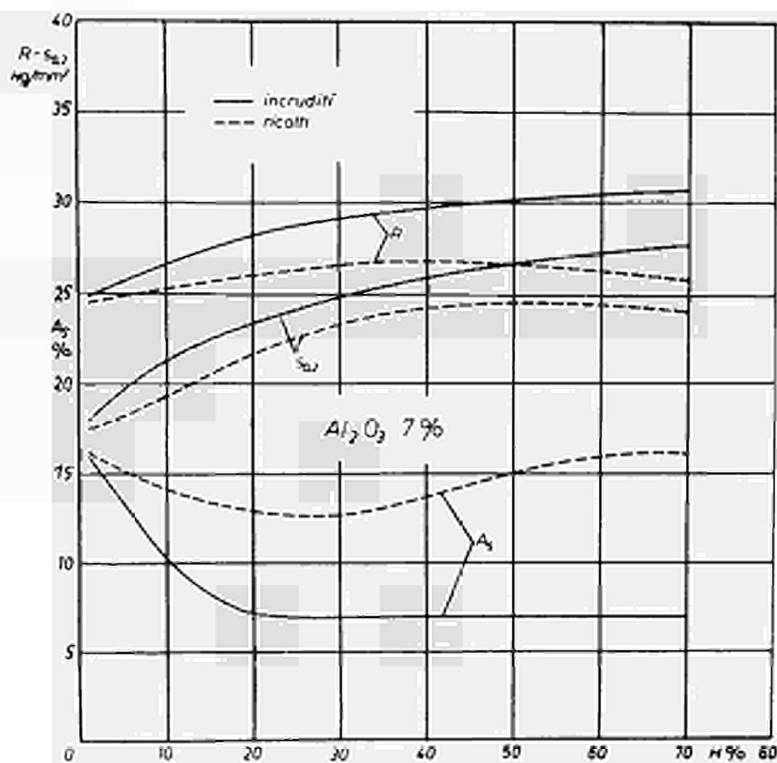


FIG. 15

Mechanical properties of sheets of SAP-ISML 7 % Al<sub>2</sub>O<sub>3</sub> vs. work-hardening, before and after (dotted lines) - annealing - at 932° F (2 hrs).

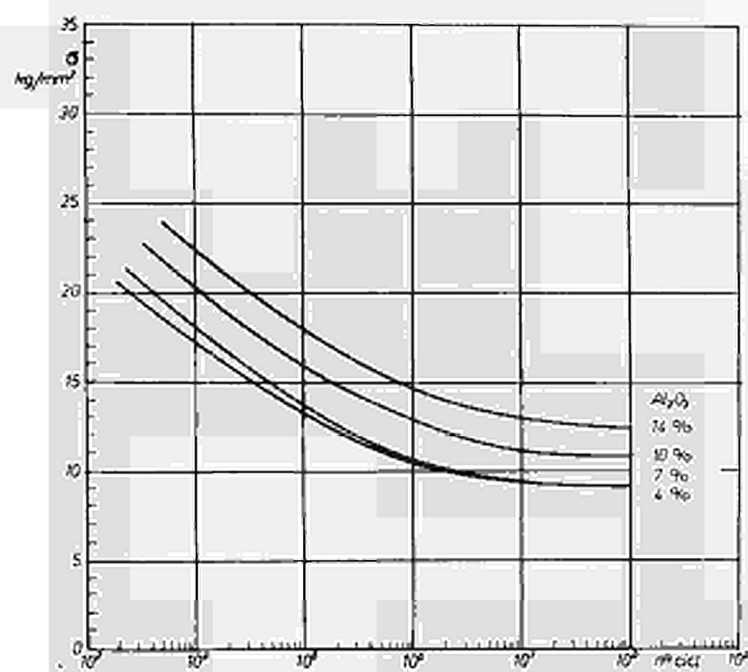


FIG. 16

Fatigue limits of SAP-ISML (without notch) at different contents of  $\text{Al}_2\text{O}_3$ .

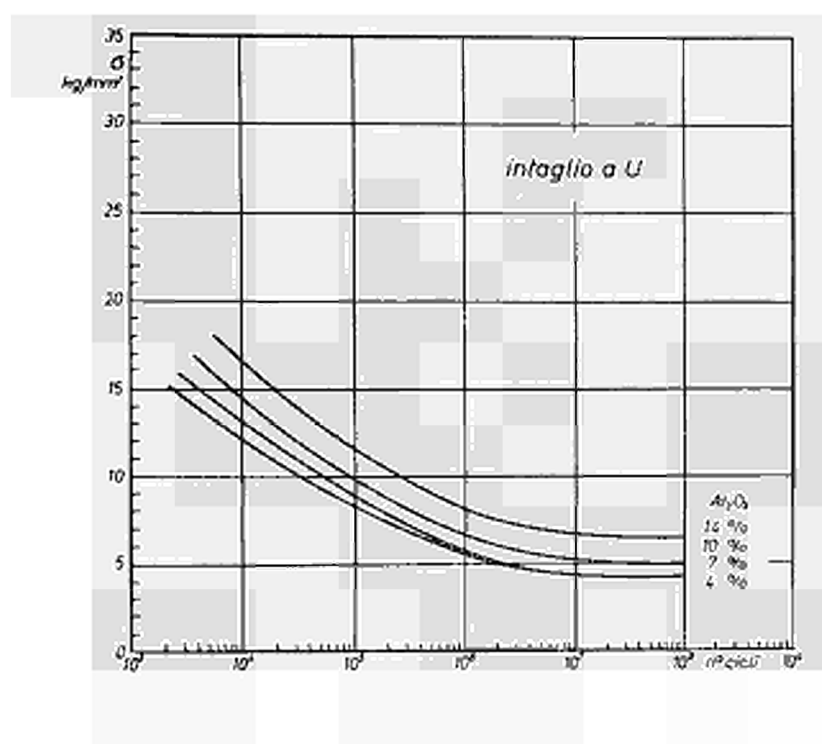


FIG. 17

Fatigue limits of SAP-ISML (with a U notch) at different contents of Al<sub>2</sub>O<sub>3</sub>.

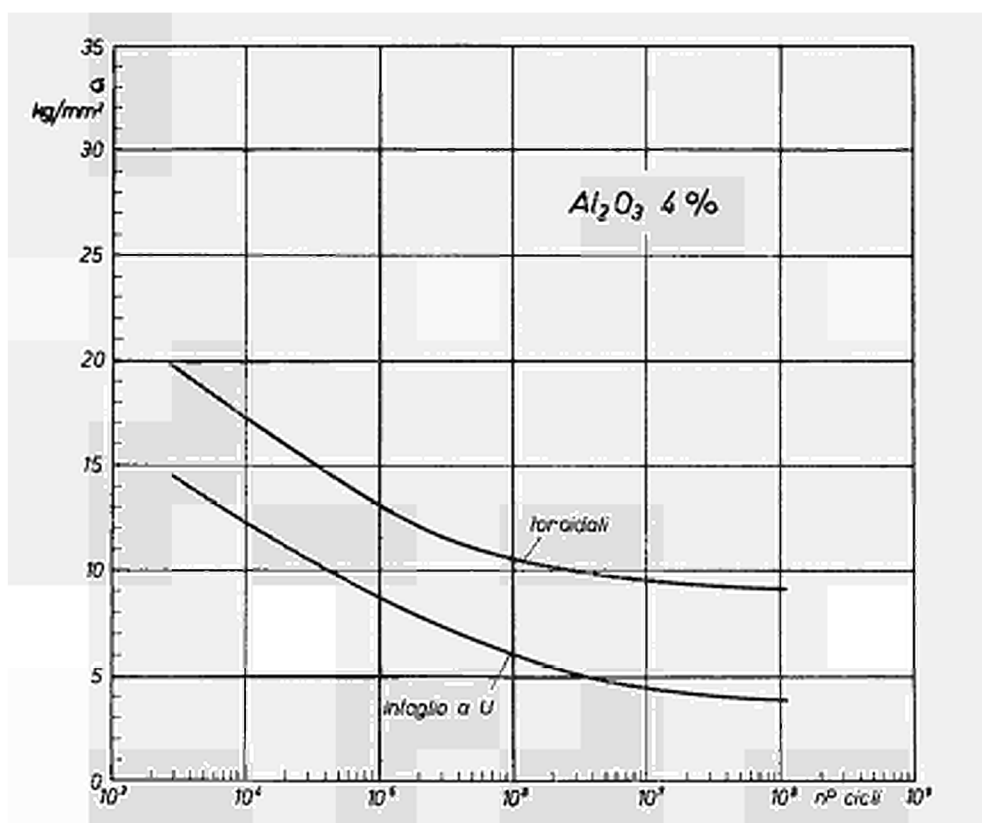


FIG. 18

Fatigue limits of SAP-ISML 4%  $\text{Al}_2\text{O}_3$ , respectively without notch and with an U or V type notch.

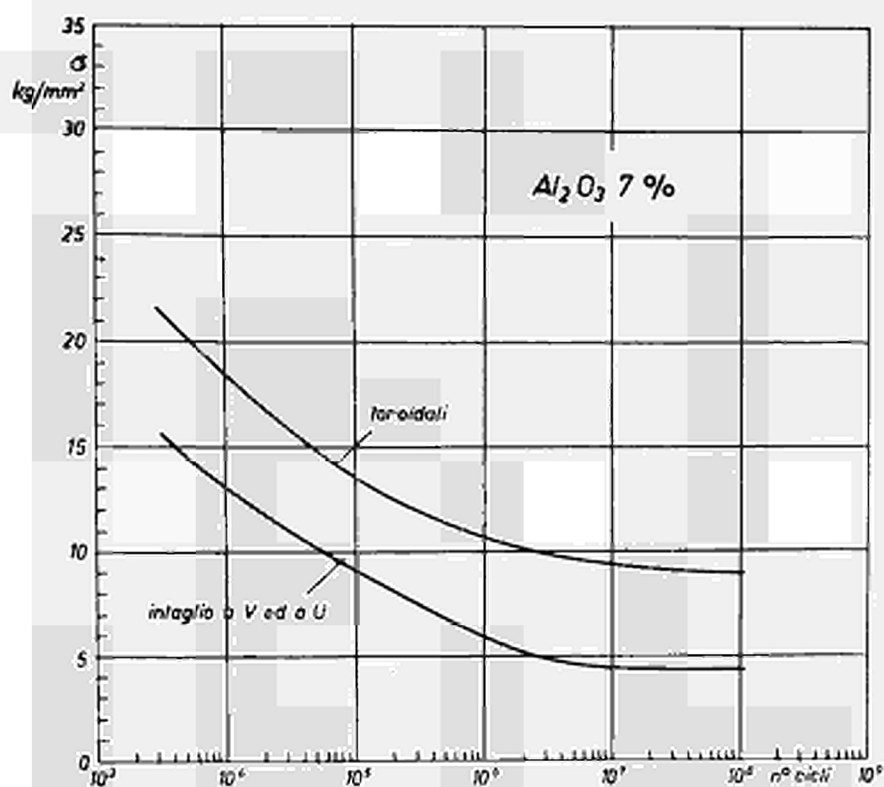


FIG. 19

Fatigue limits of SAP-ISML 7%  $\text{Al}_2\text{O}_3$ , respectively without notch and with an U or V type notch.

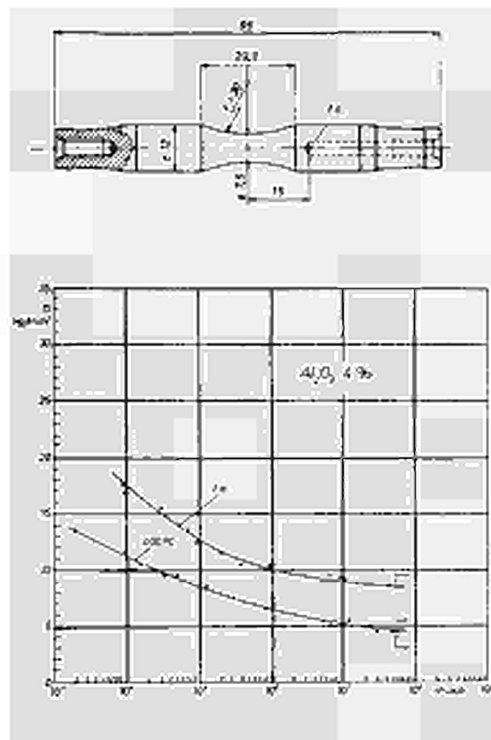


FIG. 20

Specimen used for the fatigue tests at 752° F on SAP-ISML 4 % Al<sub>2</sub>O<sub>3</sub> and results obtained.

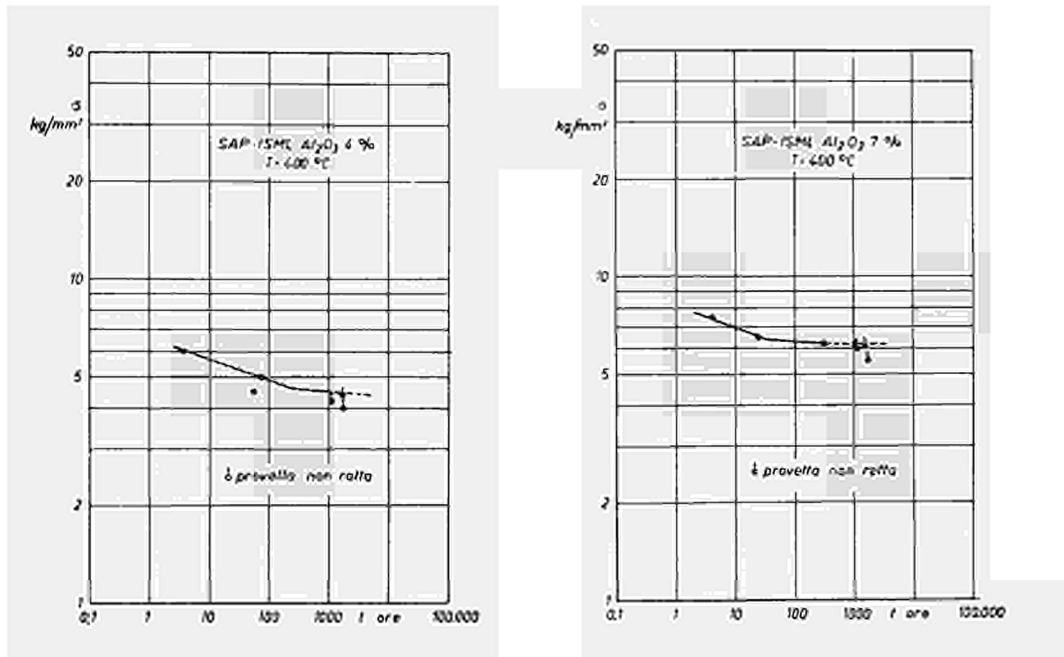
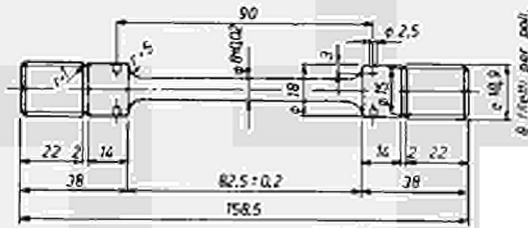


FIG. 21

Specimen used for creep tests and results at 752° F for SAP-ISML 4 % Al<sub>2</sub>O<sub>3</sub> and SAP-ISML 7 % Al<sub>2</sub>O<sub>3</sub>.

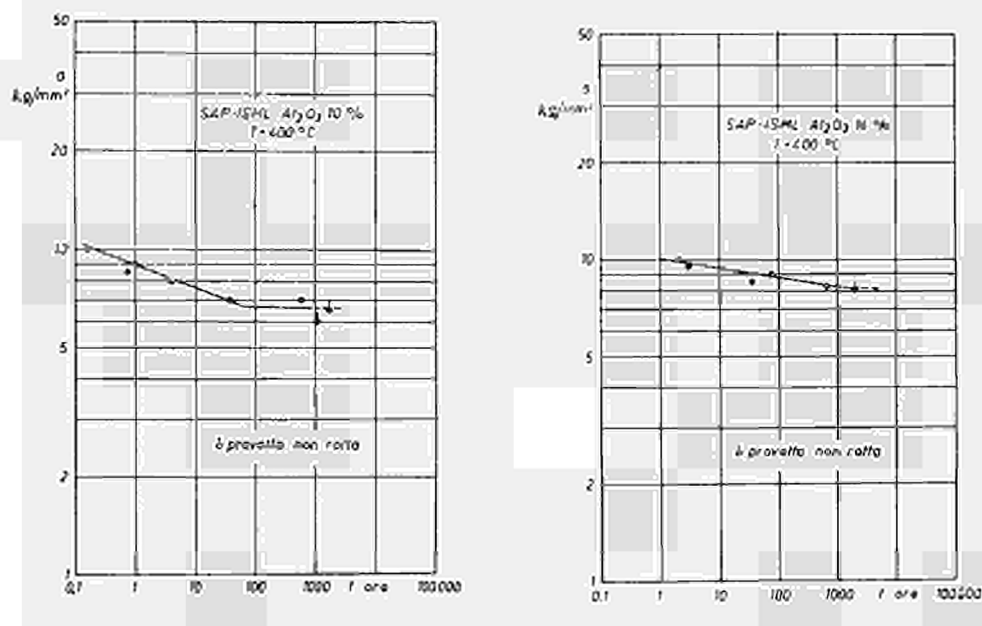


FIG. 22

Results of creep tests at 752° F for SAP-ISML 10 % Al<sub>2</sub>O<sub>3</sub> and SAP-ISML 14 % Al<sub>2</sub>O<sub>3</sub>.



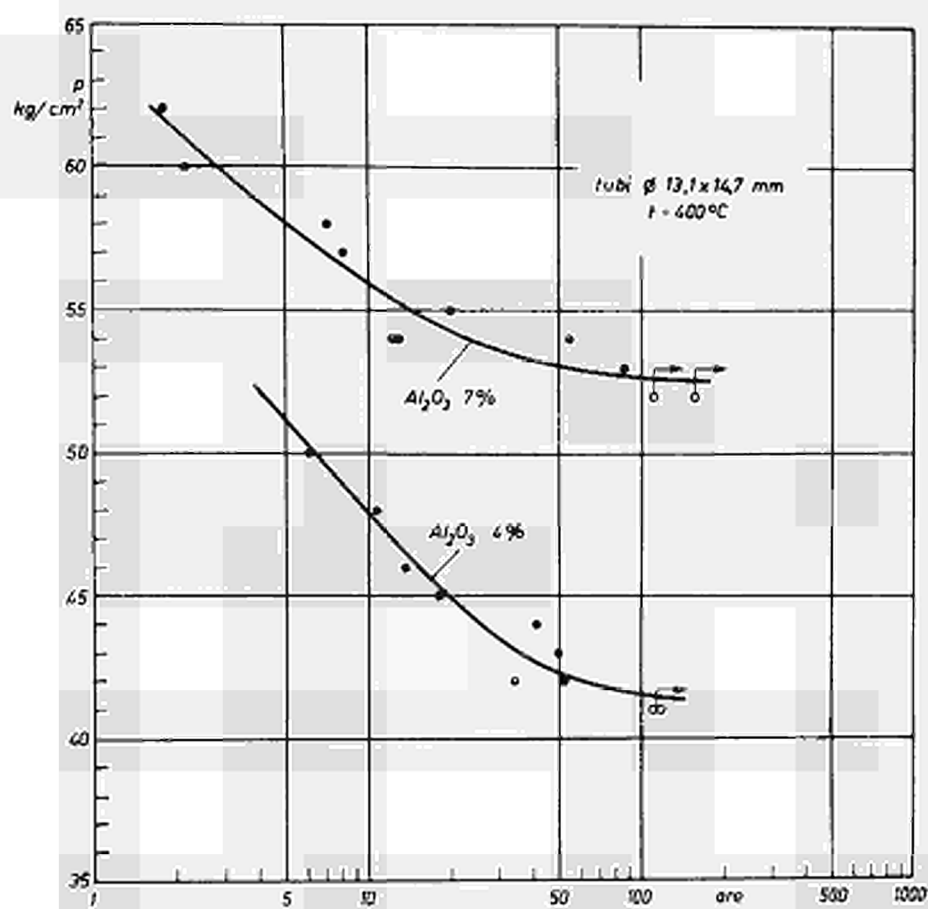


FIG. 23

Bursting pressure of tubes of SAP-ISML 4% Al<sub>2</sub>O<sub>3</sub> and SAP-ISML 7% Al<sub>2</sub>O<sub>3</sub> vs. life of specimens under pressure.



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