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EUROPEAN ATOMIC ENERGY COMMUNITY - EURATOM

**DISPERSION STRENGTHENED ALUMINIUM FOR
NUCLEAR PURPOSES, WITH PARTICULAR EMPHASIS ON
CORRELATION BETWEEN CREEP STRENGTH AND
MICROSTRUCTURE PARAMETERS**

by

D. GUALANDI*, D. GELLI*, P. JEHENSON, L. MORI* and M. PAGANELLI***

***Istituto Sperimentale Metalli Leggeri, Novara**

****Euratom**

1968



ORGEL Program

**Joint Nuclear Research Center
Ispra Establishment - Italy**

Metallurgy and Ceramics

Euratom/ISML Contract No. 065-64-7 TEOI

**Paper presented at the International Conference on the Strength of Metals and Alloys
Tokyo (Japan), September 4 - 8, 1967**

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SUMMARY

A general abstract of the mechanical properties, with reference to creep behaviour correlated with the structure parameters is presented.

KEYWORDS

SAP
MECHANICAL PROPERTIES
TEMPERATURE
FUEL CANS
TUBES
ORGANIC COOLANT
TENSILE PROPERTIES
CREEP

FATIGUE
THERMAL CONDUCTIVITY
ALUMINIUM OXIDES
ABUNDANCE
ELECTRON MICROSCOPY
PREPARATION
IMPURITIES

Internal Friction

DISPERSION STRENGTHENED ALUMINIUM FOR NUCLEAR PURPOSES,
WITH PARTICULAR EMPHASIS ON CORRELATION BETWEEN CREEP
STRENGTH AND MICROSTRUCTURE PARAMETERS (+)

1) Dispersion hardened aluminium (SAP is a typical example) has interesting mechanical properties at temperatures in the range of $350 \div 450^{\circ}\text{C}$ (ref. 1), while keeping the good nuclear properties of aluminium.

For this reason, Al-Al₂O₃ composite materials have been studied in the field of ORGEL, a nuclear reactor project elaborated by Euratom, the nuclear authority of the six European countries of the Common Market. The main characteristics of ORGEL are (ref. 2): uranium monocarbide (from natural or slightly enriched U), as fuel; aluminium hardened by dispersion with Al₂O₃ (SAP), as cladding; heavy water as moderator; organic compound with low vapor pressure (terphenyls) as heat transfer material.

2) The considerable metallurgical problems originated by the use of a material like SAP, which has to withstand temperatures of about $400 \div 450^{\circ}\text{C}$, while the melting point of aluminium is 660°C , have been treated in the EURATOM Research Center (J. R. C.) in Ispra and through contracts with the Italian company Montecatini-Edison, by the Nuclear Service of this company and the Istituto Sperimentale Metalli Leggeri (I. S. M. L.).

A group of different contracts was negotiated, starting in 1960, by these companies and EURATOM, in order to study the different aspects of the problem (properties, powders, technical improvement, industrial production, basic research, control methods, etc.).

In this paper, we present a general abstract of the mechanical properties, with reference to creep behaviour correlated with the structure parameters.

(+) Manuscript received on June 20, 1967.

3) Materials used

During the initial part of the research, it was necessary to improve the quality of the semifinished products from the point of view of heat stability.

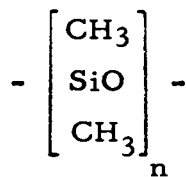
That was obtained through a vacuum heat treatment, which stabilizes the oxide and lowers the residual content of hydrogen to values of about 1 ppm (ref. 3).

The materials, vacuum sintered through this ISML patented process, were called SAP-ISML.

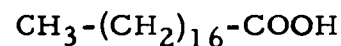
At the same time, the SAP powders were also improved, reducing the Fe and Si contents and checking their homogeneity and quality, through the use of new specifications and controls given to the producer (ref. 4, 5).

The material was further improved lowering to the absolute minimum the iron content (less than 0.01^w%), using pure aluminium (Raffinal:Al = 99.99%) in place of normal aluminium and manufacturing the powder with special equipment in order to keep such a high purity; this purer oxidized material was patented and received the name PUROXAL. During the fabrication of Puroxal and also of SAP or SAP-ISML, namely during the milling for oxidation of the atomized Al powder, an addition of stearic acid is necessary, as lubricant; as a consequence, the finished product contains some aluminium carbide (Al₄C₃). In order to avoid as much as possible the presence of this non-metallic impurity, a special kind of Puroxal was manufactured, in which a silicone compound was used instead of stearic acid.

Silicone compound



Stearic acid



The silicone compound contains less carbon than stearic acid. Silicon can be accepted from the nuclear point of view and is not harmful from a metallurgical point of view.

The special Puroxal, made with a silicone compound in place of stearic acid during the milling of the powder, is called Puroxal-S in this paper.

Another material in which the starting material was an alloy of aluminium and magnesium with 2-5% of Mg has been tested. The purpose of this modification was to change the work hardening characteristics of the aluminium matrix through the solution of Mg and to change the dispersed phase from Al_2O_3 to other mixed oxides. This material will be called PUROXAL-M in this paper.

Patents are pending concerning Puroxal-S and PUROXAL-M products. See also note at the end of the paper.

In the following table, the main chemical features of some typical products are listed:

Type	Fe w%	Si w%	C w%	H ₂ ppm	Al ₂ O ₃ w%	MgO w%
SAP	0,2	0.15	0.25	10-20	≈ 7	-
SAP-ISML	0.1	0.06	0.25	1-2	≈ 7	-
PUROXAL	0.01	0.005	0.25	1-2	≈ 10	-
PUROXAL-S	0.01	0.10-0.15	0.1	1-2	≈ 10	-
PUROXAL-M	0.01	0.10-0.15	0.1	1-2	≈ 4	≈ 2

4) Structure

The structural parameters of Al-Al₂O₃ composites of the type given above are divided in 2 categories:

- those affecting the Al matrix,
- those affecting the dispersed phase.

4.1) Matrix structural parameters

Figs. 1-2 (optical micrographs) and fig. 3 (electron micrograph, in transmission: X 50,000) show the general structure of the 5 materials:

SAP
SAP-ISML
PUROXAL
PUROXAL-S
PUROXAL-M

Optical micrographs can only indicate the overall homogeneity of the oxide distribution: the dimensions of the Al_2O_3 particles and of the grains can be measured from the electron micrographs. The dimensions of the sub-grains are not changed by a heat treatment for a long time even at temperatures close to the melting point, and this is the basis of the good mechanical properties at high temperature of these composite materials.

The boundaries between the grains are rich in dislocations, anchored to clusters of the dispersed oxide phase particles or to individual particles of proper size of this dispersed phase. The quantity of this phase is proportional to the oxide content and consequently, generally speaking, there is an inverse proportionality between the oxide content (i. e. chemical composition) and the average diameter of the grain (see table I, for the average dimensions).

The very fine oxide particles sometimes do not show a good efficiency for blocking the dislocation walls.

Another feature of the matrix is the presence of microcracks with different dimensions but normally in the range between 2 and $10/\mu$. They are divided according to size and their number is given per mm^2 of the observed replica of the surface of the specimen.

These microcracks are located along the alignments of oxide particles; in the creep process the presence of these microcracks, inherent in the material, is very important.

All materials tested show the presence of this structural feature: from table II it is possible to observe that Puroxal has less microcracks than SAP.

Dislocations inside the grains

The density of dislocations in the interior of the grains in this type of materials is normally not high, as can be seen from the typical structure taken at the electron microscope, shown previously in fig. 3.

In Puroxal-M, it has been found sometimes a dislocation network in the interior of the grains (fig. 4).

4. 2) Structural parameters affecting the dispersed phase

The dimensions and the shape of the particles of the dispersed phase are the first of the structural parameters of this second group. For SAP, SAP-ISML, PUROXAL and PUROXAL-S these phases are γ Al_2O_3 and χ Al_2O_3 , shaped as platelets of 0.01 micron thickness and 0.1 micron average width.

Some of these platelets can be grouped in clusters, of about 1-2 microns in average diameter. There is consequently a "factor" of homogeneity of the oxide platelets. Puroxal products have finer platelets and are more homogeneous as far as the oxide distribution is concerned.

When instead of pure aluminium, an alloy Al-Mg (2-5 w% magnesium is the range tested), is atomized, oxidized and sintered, a different oxide is present, with a similar dispersion but with tridimensional particles, not platelets (fig. 5); X-ray diffractometry and chemical analysis show these particles to be constituted by a spinel (Al_2O_3 -MgO) and MgO, without Al_2O_3 .

It is interesting to note that the oxidation of Mg takes place not only during the milling of the powder, accomplished at room temperature or a little above room temperature, but continues during sintering at 600°C, by reduction of Al_2O_3 .

Magnesium atoms migrate to Al_2O_3 particles sites and there the reaction $\text{Al}_2\text{O}_3 + 3 \text{Mg} \longrightarrow 3 \text{MgO} + 2 \text{Al}$ takes place.

A certain amount of free magnesium can react with the silicon derived from the lubricating silicone compound to form Mg_2Si . The dispersed phases can

consequently be: (Al_2O_3) , MgO , $MgO \cdot Al_2O_3$, Mg_2Si ,

Given the diffusion coefficients of Mg and oxygen, the most valid hypothesis is that MgO is formed in situ, where formerly were the platelets of Al_2O_3 .

Other phases present in all materials are AlN and Al_4C_3 , which respectively take origin from the reaction of the powder with the nitrogen of the atmosphere (reaction very active above $500^\circ C$) and with the carbon present in the stearic acid (or, in less quantity, in the silicone compound).

In the presence of Mg also a little quantity of magnesium carbide or nitride can probably form.

5) Mechanical properties of Al- Al_2O_3 finished products

Before taking into special consideration the creep properties of the different types of Al- Al_2O_3 composites, it is useful to give an idea of the general mechanical properties of these materials: there are a few general patterns which are quite characteristic of the mechanical behaviour of the composites and can be so summarized:

1. at a fixed temperature, the ultimate tensile strength and the yield strength rise with the oxide content. Ductility, as measured by total elongation, is inversely affected by the increase of the oxide content.
From the structural point of view, higher oxide content can block the boundaries of the sub-grains to a stable smaller size.
2. What is said above is valid for extruded shapes obtained by hot transformation; when, via cold work, the grain is subsequently more fragmented, the behaviour changes as follows: at room temperature, the strength is higher (and the elongation lower) in the cold worked specimens in comparison with the hot extruded material. At high temperature however, the reverse is true: mechanical strength is lower in the cold worked state with respect to the extruded shapes of corresponding size with no apparent change in elongation.

The effect of cold work (improvement of mechanical properties at room temperature, and worse properties at high temperature) is structurally

understood as due to the microcracks, which are much larger and in greater number in cold worked specimens.

3. When the values of UTS (or YS) are plotted against the corresponding values of elongation, in conventional aluminium, the points fall on the same curve regardless of the test temperature.

In SAP or PUROXAL the points group on different curves, (e.g. one curve for room temperature and one for elevated temperature) suggesting a different behaviour of the material under test at the two different temperatures. From the structural point of view, this also may be referred to the different influence, at different temperatures, of the grain-boundaries and of the microcracks existing in the matrix.

4. Another peculiar feature of Al-Al₂O₃ composites in comparison with conventional Al-alloys is the fact that at every level of oxide content, the time to rupture (i.e. the rate of straining the specimen) has at room temperature a rather limited effect on total elongation and, at high temperature a very marked influence on this property (fig. 6).

6) Creep properties

Concerning creep, these materials show the following general characteristics:

1. in SAP the curve of elongation vs time does not show evidence of tertiary creep (fig. 7). After the initial deformation the creep curve continues with a very small slope to the point of rupture of the specimen;
2. the slope of this portion of the creep curve is practically constant and very small, denouncing a limited deformability of the small grains;
3. the lines which give the stress necessary to bring about a certain deformation (say 0.2%, 0.3%, 0.4%) are almost parallel, very close to each other and close also to the line which gives the stress to rupture vs time to rupture (fig. 8);
4. the resistance to creep for materials in the range 4-14% Al₂O₃ in the as extruded state, given as the stress which produces the rupture in 1000 hrs at 400°C (see fig. 9) is about 70% of the UTS at the same temperature;
5. the stress to rupture does not become negligible (as would be the case in all other aluminium alloys) even at a temperature (646°C), very close to the melting point of aluminium (see fig. 10).

It is interesting to note that even at such a high temperature the total deformation after the creep test is very small: namely, the material preserves its rigid structure.

7) Creep behaviour of Puroxal and Puroxal-S

A group of creep curves obtained at 450°C on a 10% Al₂O₃ Puroxal is shown in fig. 11.

A similar group of curves for a 10% Al₂O₃ Puroxal-S (i. e. obtained with a silicone compound as lubricant during the milling of the powder) is shown in fig. 12. The comparison of the results indicates that the creep strength of Puroxal-S is somewhat reduced but shows an elongation that reaches 1% for a life of the specimen up to 50 hrs. SAP, in these conditions, has only 0.2% (see also fig. 13).

8) Creep behaviour of Puroxal-M

Puroxal-M is the name conventionally given to the material when, instead of pure aluminium, the starting material is an Al-Mg alloy, with 2÷5% magnesium (see note in appendix).

The different processes leading to these materials (Puroxal, Puroxal-S, Puroxal-M) have been described in the corresponding applications for patents.

A group of creep curves for a sintered material of this last type, with 2% Mg, is shown in fig. 14. The stresses leading to rupture in a given time are not reduced in comparison with Puroxal or Puroxal-S when keeping in mind the oxide contents, but the elongations in the range of 1-500 hrs are much higher than in all previously described materials: namely it is possible to have over 10% elongation with 1 hr life of the specimen, and over 5% with 50 hrs of time to rupture.

When the life of specimen is over 500 hrs the elongation is reduced to small values, 0.3÷0.5%, still somewhat higher than in SAP.

9) Structural aspects of creep

9.1) Influence of grain size

The grain size, as observed in electron micrographs, has been found directly correlated with the tensile strength and consequently the creep strength. In extruded rods of SAP-ISML the correlation between oxide content and average

dimensions of the grain is given in fig. 15.

Puroxal has finer particles of Al_2O_3 and the grain is, with the same oxide content, appreciably larger as shown in the same fig. 15.

This can explain the reduced mechanical and creep strength observed.

9.2) Influence of microcracks

Microcracks are never found on areas free of Al_2O_3 particles and are more frequent in the regions where said particles form clusters.

They have normally their larger axis in the direction of extrusion and it is assumed that the interface between the oxide particles and the aluminium matrix favours the presence of microcracks.

The influence of microcracks is particularly evident in ductility at elevated temperature as shown in table III.

During the creep test the influence of microcracks is at its maximum because during the test itself new microcracks form and those previously present, resulting from fabrication, grow in dimensions; as shown by observations at the electron microscope.

The best performance of Puroxal-M with regard to elongation is due to the markedly reduced number of microcracks.

10) Influence of the grain boundaries

The typical structure of SAP and Puroxal given previously, clearly shows that the boundaries of the grains are dislocation walls which presumably can not have the same mechanical properties of the interior of the grains, in which hardening particles of Al_2O_3 are present. The higher strength of the interior of the grains with respect to the boundary is a condition, as it is well known, leading to intercrystalline fracture and reduced elongation.

It is also assumed that the boundary of the grain can show a "quasi viscous" behaviour (ref. 6) in the sense that, at constant temperature, the strength of the grain (σ_c) can be assumed as constant with the rate of strain (fig. 16),

while the shearing strength (γ) of the boundary varies with the rate of strain as shown in the same figure.

The overall influence of the rate of strain on elongation will be, as a consequence, of the type shown at the bottom of said fig. 16.

As the rate of strain in creep tests is small, the influence of a quasi-viscous behaviour of grain boundaries is towards a reduction of the elongation.

11) Conclusions

The experimental data presented can not bring to a general conclusion on creep behaviour of Al-Al₂O₃ composites but the following points appear established:

- a) the high mechanical strength at elevated temperature of the composites examined is mainly correlated with the very reduced grain size, stable at high temperature, due to the presence of the dispersed phase;
- b) when comparing SAP material and PUROXAL material with different composition in oxide content, but with the same grain size, the mechanical strength is similar in the two materials;
- c) the strength of the grain boundaries is reduced in comparison with the strength of the grains and the behaviour of the boundary with the rate of strain is such as to give smaller elongation when the rate of straining is reduced. Consequently, in creep tests reduced elongations are to be expected;
- d) the presence of microcracks in the structure of these composites, which can grow in number and dimensions, at elevated temperature under a stress producing a strain, is another factor that leads to rupture with a very reduced elongation;
- e) the improvement in the material obtained with the transformation of the dispersed phase from Al₂O₃ to a mixed oxide (MgO + MgO Al₂O₃), has been a marked increase of creep ductility when the life of the specimen does not exceed 500 hrs; in normal SAP materials, a relative ductility can not be maintained over 1 hr, or even less. From a practical point of view then, this limited achievement can be of a notable interest when the material has to withstand a limited overstress (thus an overstrain), during a limited time; this often arrives during the use of a fuel cladding.

APPENDIX

The name Puroxal is protected by a trade mark; in order to distinguish from it the products in which the starting powder is an aluminium-magnesium alloy, to these finished products (containing as dispersed phases a mixture of MgO and MgO . Al₂O₃) the general name ALMOX will be given, followed, if necessary, by letters and figures. For example ALMOX 6-4 would signify an oxidized Al-Mg alloy, with 6% Al₂O₃ and 4% MgO in the finished product.

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TABLE I - AVERAGE GRAIN DIMENSIONS IN EXTRUDED RODS OF SAP-ISML VS CONTENT OF OXIDE (TRANSVERSE SECTION)

Al ₂ O ₃ content %	TEMPER		
	As Extruded	After 3 hrs at 600°C	After 3 hrs at 650°C
3.8	1.1 /μm	1.1 /μm	1.1 /μm
8.5	0.6 "	0.6 "	0.6 "
10.4	0.5 "	0.52 "	0.57 "
13.9	0.45 "	0.45 "	not determined

TABLE II - NUMBER OF MICROCRACKS PER MM² IN SAP-ISML AND PUROXAL-S IN UNSTRESSED AND STRESSED SPECIMEN

Material	Unstressed Specimen			Creep Stressed Specimen			Creep Conditions		
	5 ÷ 10/μ	2 ÷ 3/μ	≤ 1/μ	5 ÷ 10/μ	2 ÷ 3/μ	≤ 1/μ	kg/mm ²	Life hrs	Temperature °C
Puroxal-S (4% Al ₂ O ₃)	0	327	404	218	182	655	2.25	501	450
Puroxal-S (7% Al ₂ O ₃)	0	312	364	309	382	673	2.75	140	450
SAP-ISML (4% Al ₂ O ₃)	36	255	629	273	255	805	4.6	1152	400
SAP-ISML (7% Al ₂ O ₃)	36	266	218	109	946	1165	5.9	1200	400

TABLE III - CORRELATION BETWEEN MECHANICAL PROPERTIES AND MICROCRACKS
IN PUROXAL-S

Mechanical Properties						Microcracks per mm ²		
20°C			450°C			5 ÷ 10	~ 2	≤ 1
UTS kg/mm ²	YS kg/mm ²	EI %	UTS kg/mm ²	YS kg/mm ²	EI %	/u m	/u m	/u m
32.8	29.3	10.2	6.75	6.3	2.0	122	410	910
31.4	26.3	12.7	7.2	6.5	9.8	0	30	127

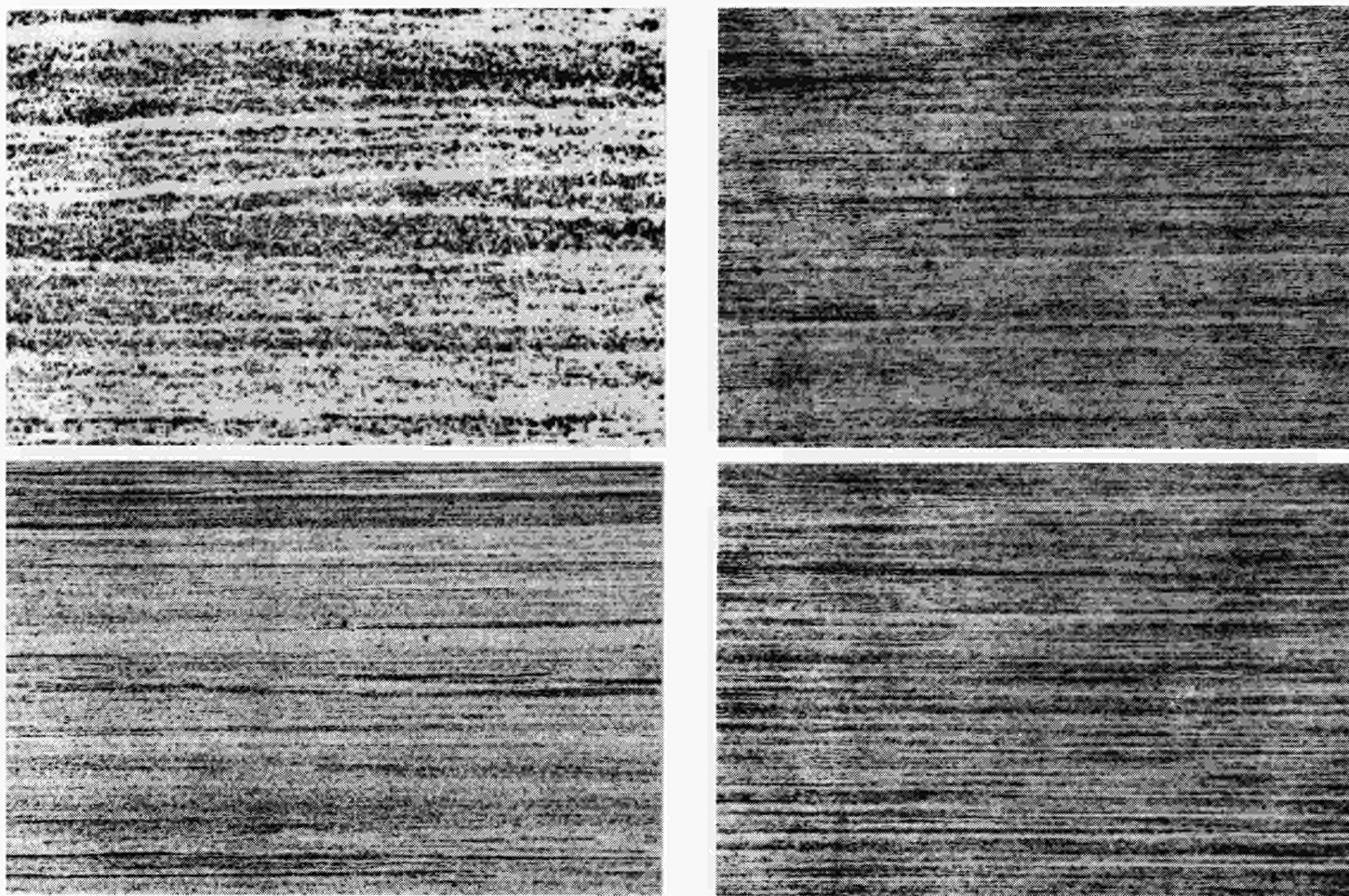


Fig. 1 - Typical microstructures (x 100) of SAP (top, left), SAP-ISML (top, right), PUROXAL (bottom, left) and PUROXAL-S (bottom, right). (Lastre N. 33831, 35709, 37507, 38504) (X 100 - Etching: HF 0.5%).

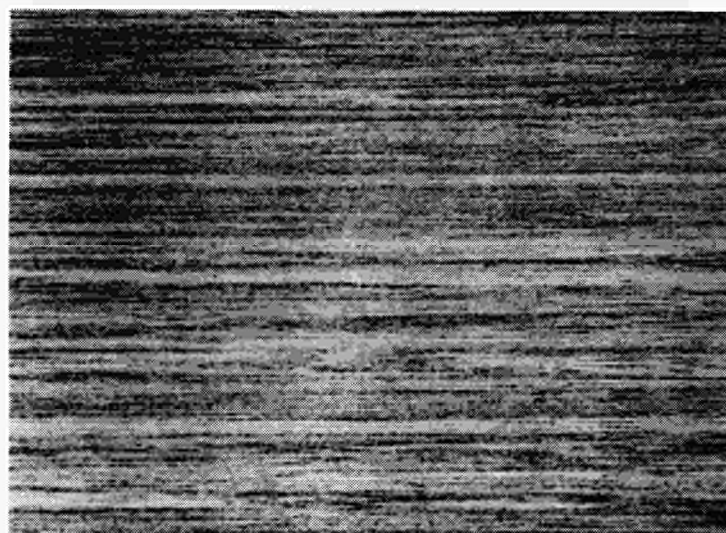


Fig. 2 - Microstructure of Puroxal-M (x 100 - Etching: HF 0.5%)
Lastra N. 41.650)

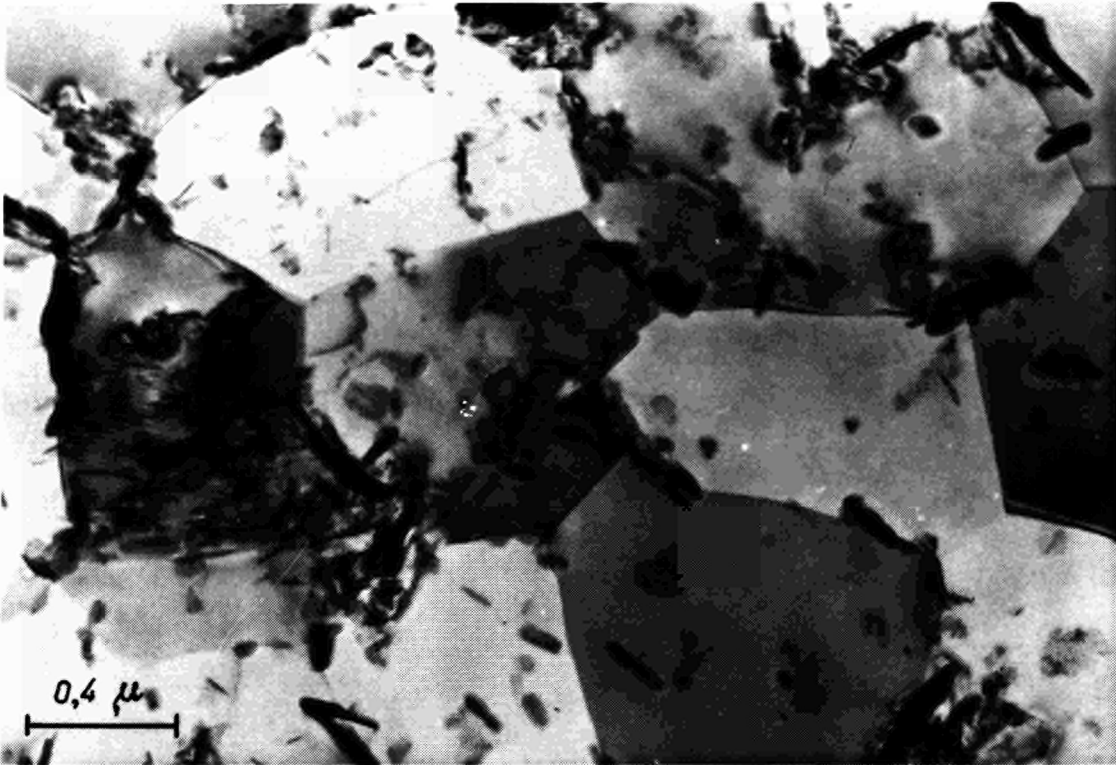


Fig. 3 - Typical microstructure taken by transmission at the electron microscope. (Plate N. 2794 el.) (x 50,000)

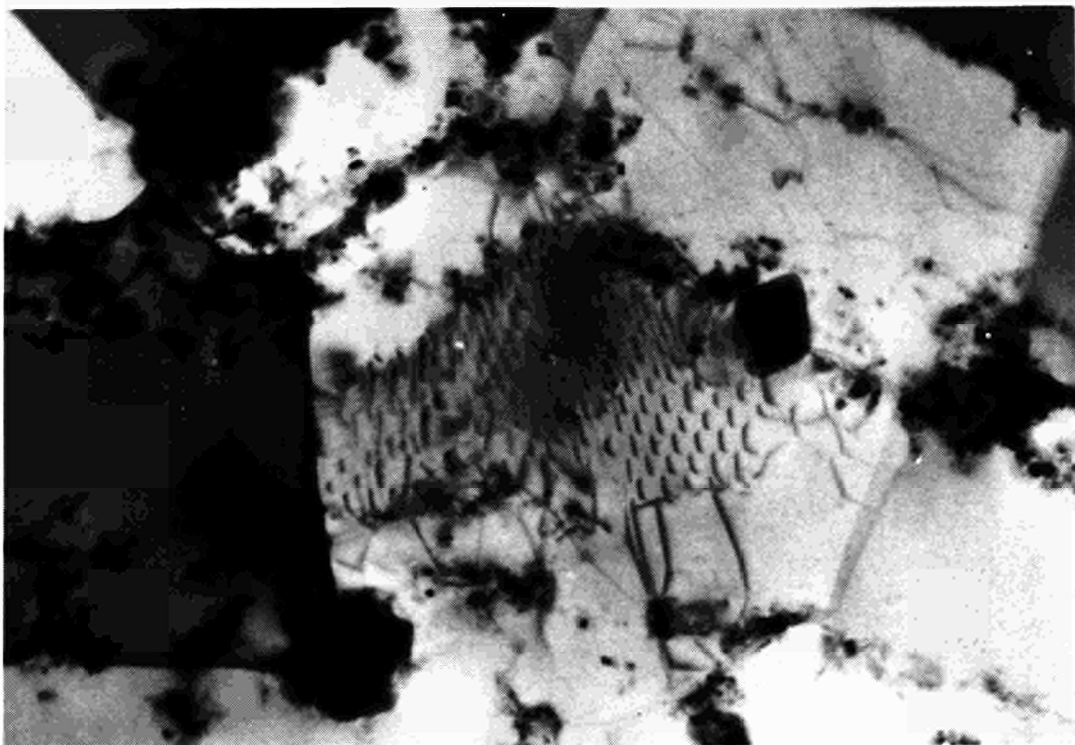


Fig. 4 - Microstructure of Puroxal-M with a dislocation net within the grain. (x 50,000) (Plate N. 5650).

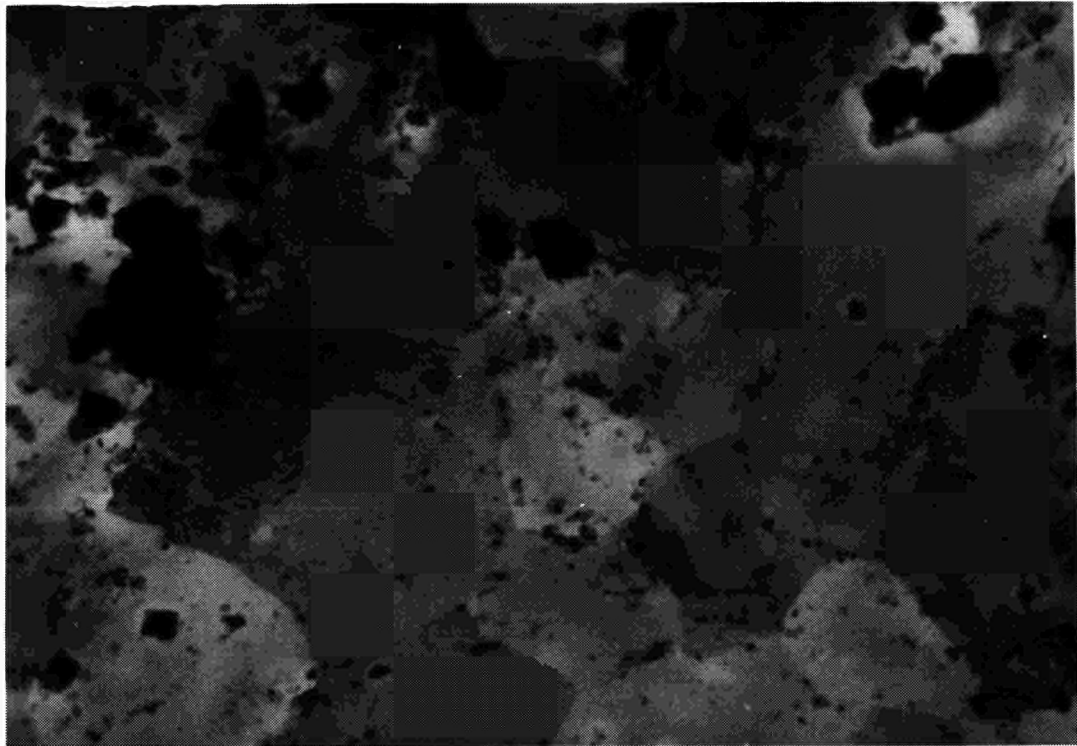


Fig. 5a - Typical microstructure of Puroxal-M taken by transmission at the electron microscope. (x 20,000)(Plate N. 5644)

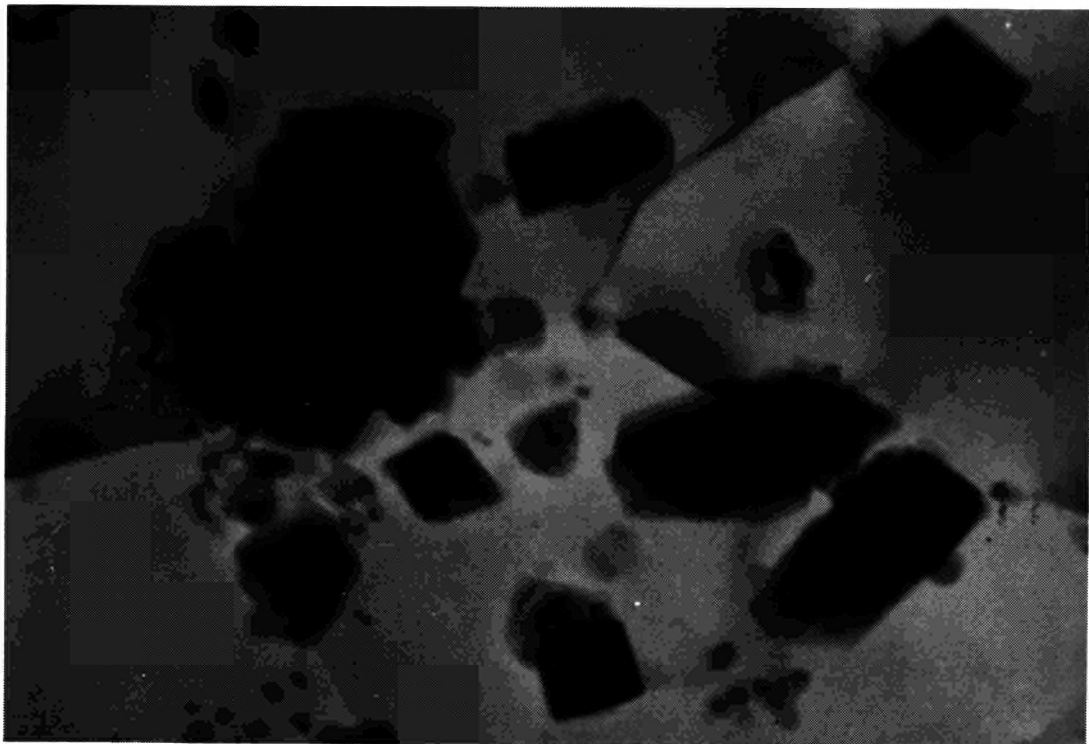


Fig. 5b - Microstructure of Puroxal-M at higher magnification. (Plate N. 5614)

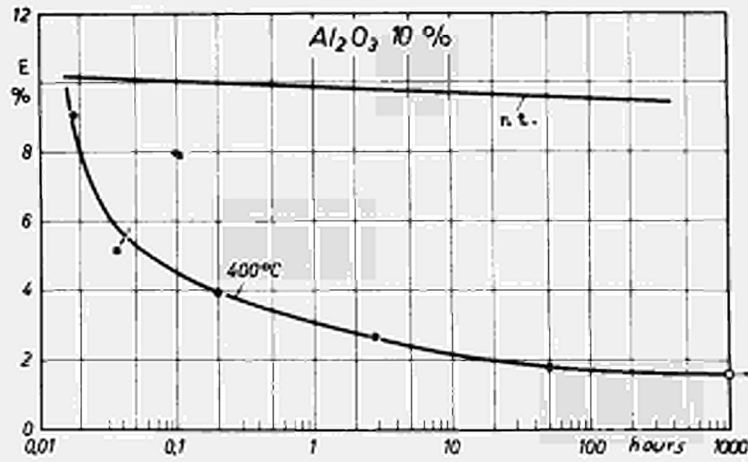


Fig. 6 - Elongation vs. life of specimen.
(Plate N. 31131/3)

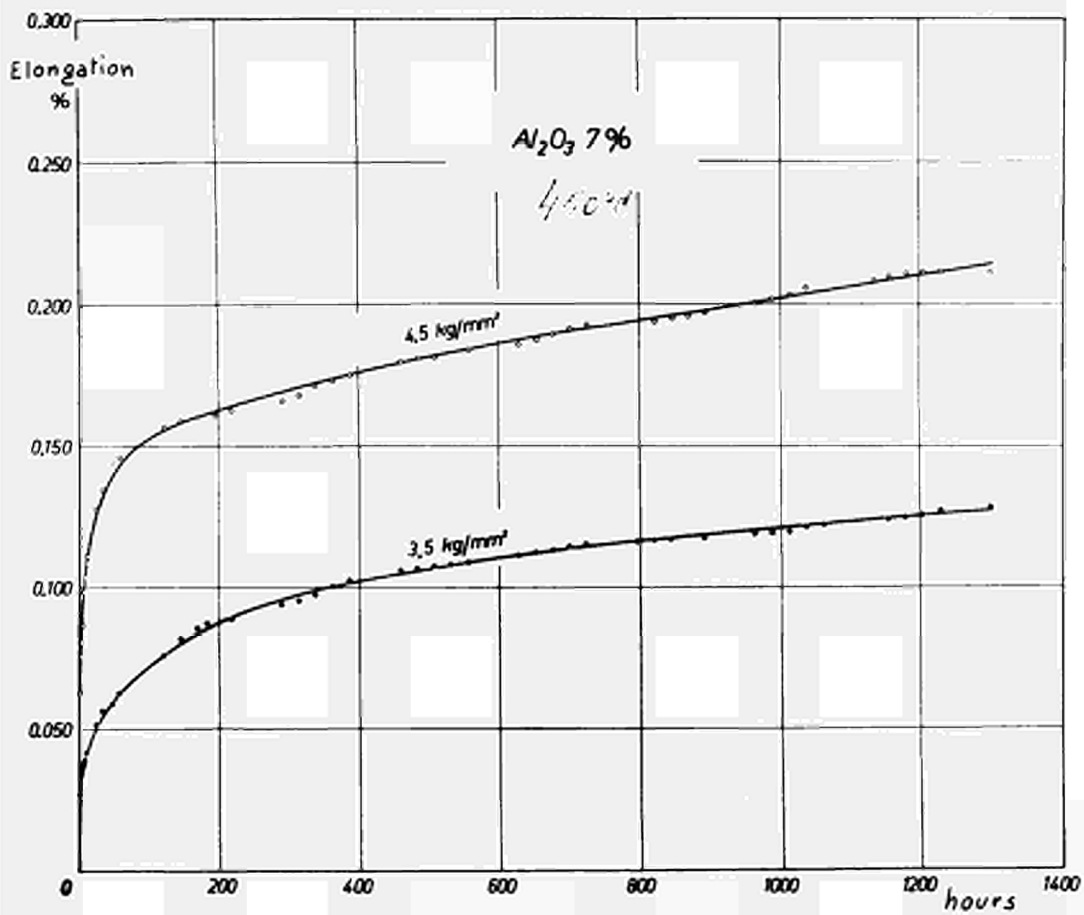


Fig. 7 - Typical creep curves with different stress of SAP 7% Al_2O_3 .
(Plate N. 5176/G)

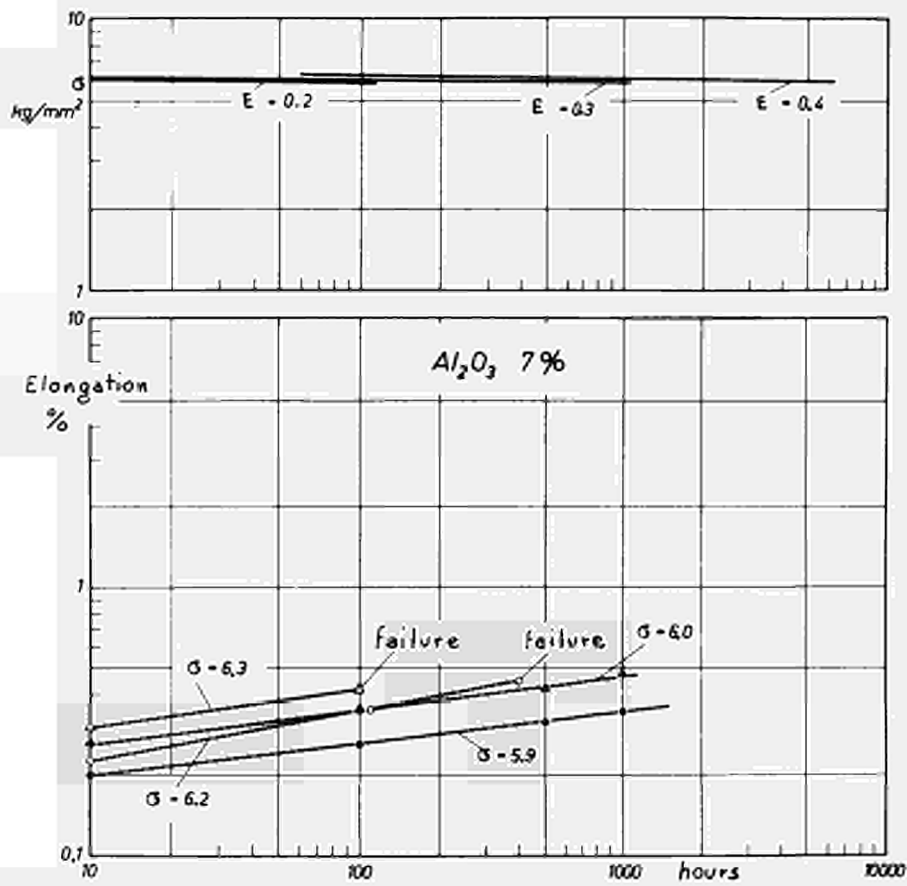


Fig. 8 - Behaviour under creep conditions at 400°C.
(Plate N. 31114/3).

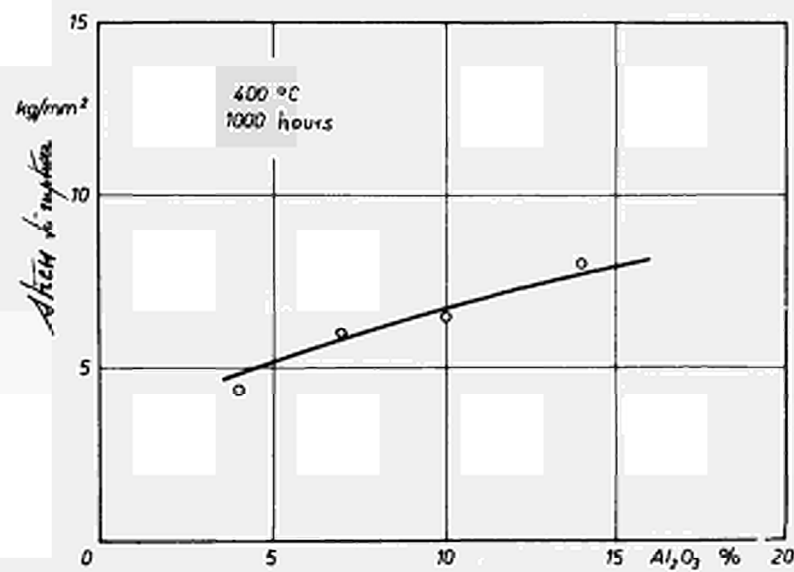


Fig. 9 - Stress producing rupture in 1000 hrs at
400°C vs. % Al_2O_3 . (Plate N. 31121/2)

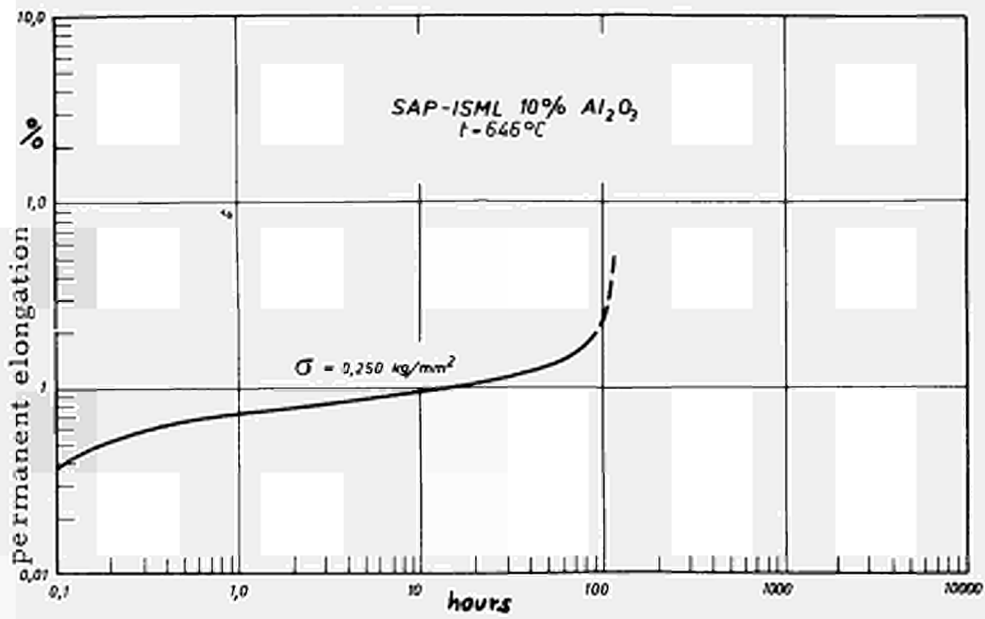


Fig. 10 - Creep curves of SAP-ISML 10% Al_2O_3 at $646^\circ C$.
 (Plate N. 31122/8).

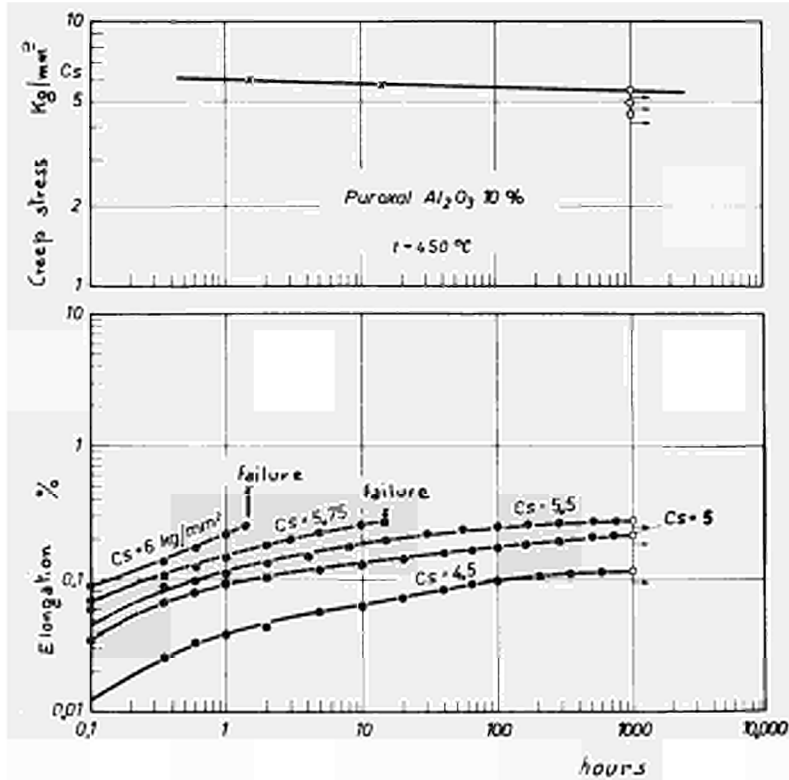


Fig. 11 - Creep curves of Puroxal (10% Al_2O_3) at $450^\circ C$.
 (Plate N. 31162/5).

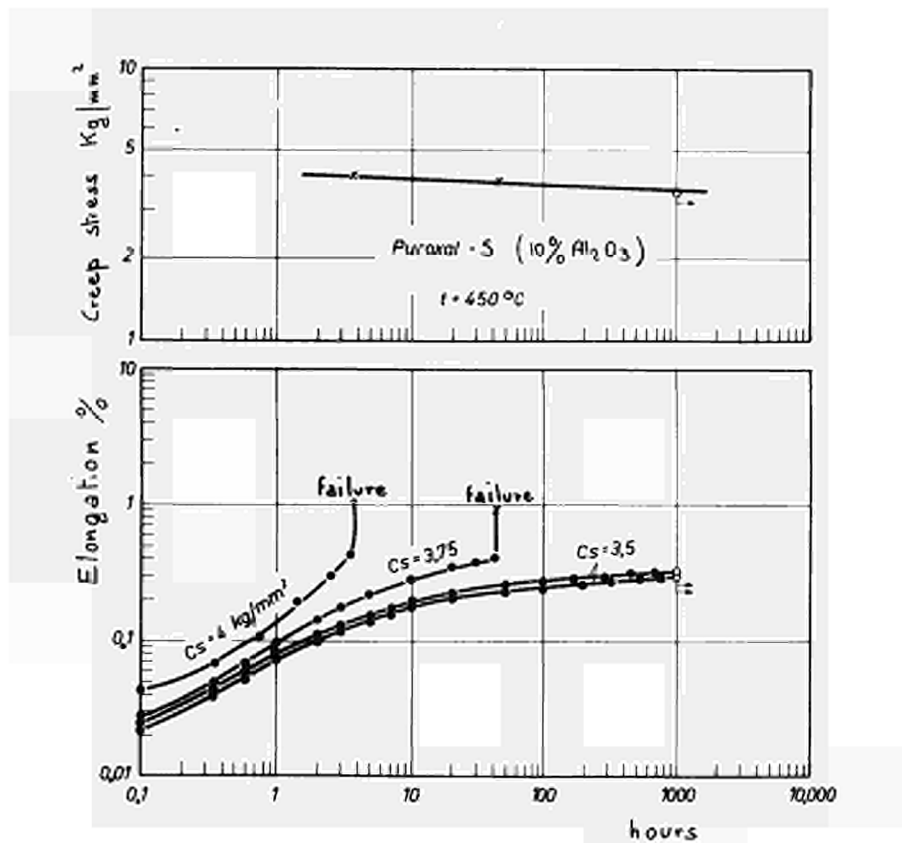


Fig. 12 - Creep curves of Puroxal S (10% Al_2O_3) at 450°C .
(Plate N. 31162/6)

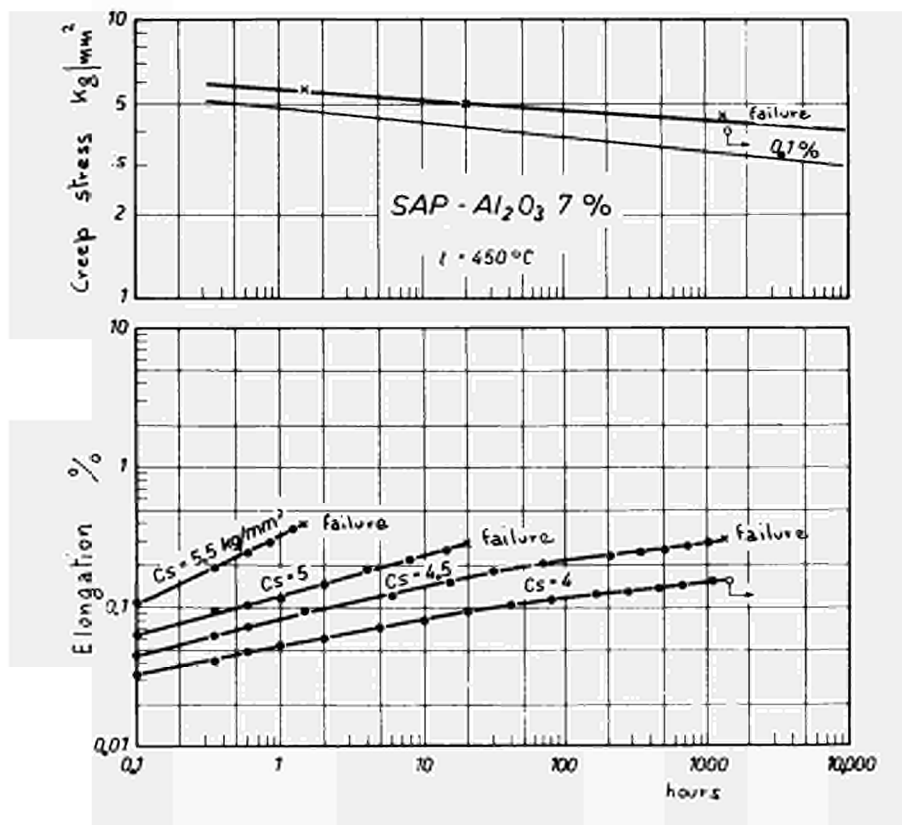


Fig. 13 - Creep curves of SAP-ISML (7% Al_2O_3) at 450°C .
(Plate N. 31156/10).

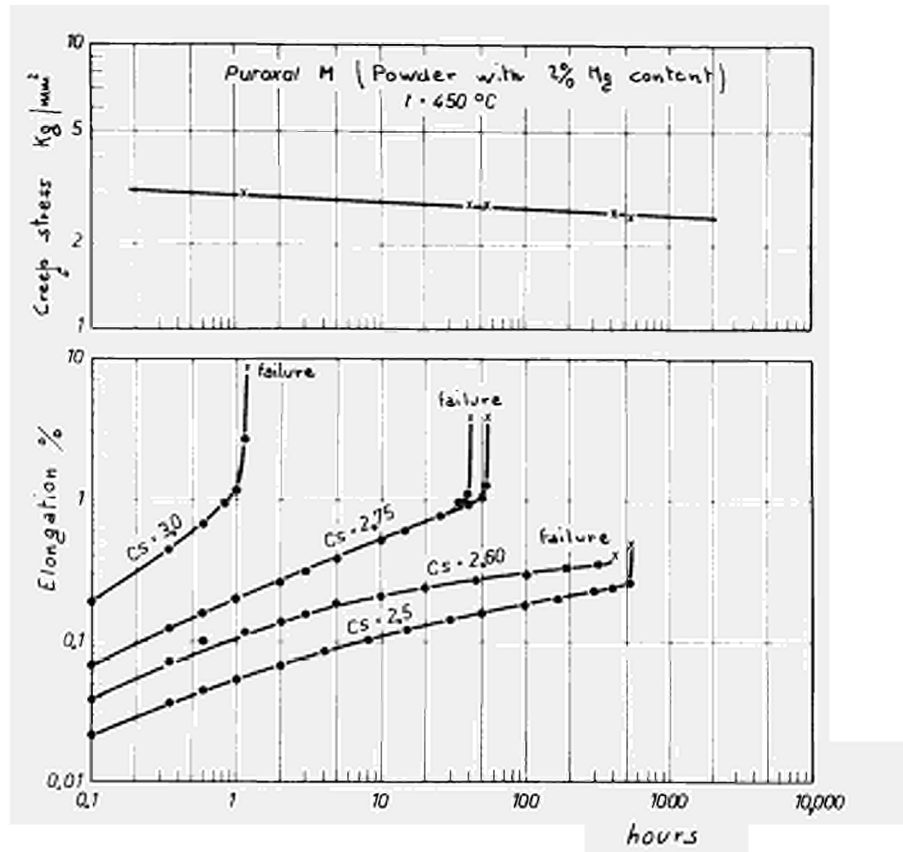


Fig. 14 - Creep curves of Puroxal-M (starting powder with 2% Mg content). (Plate N. 31160/9).

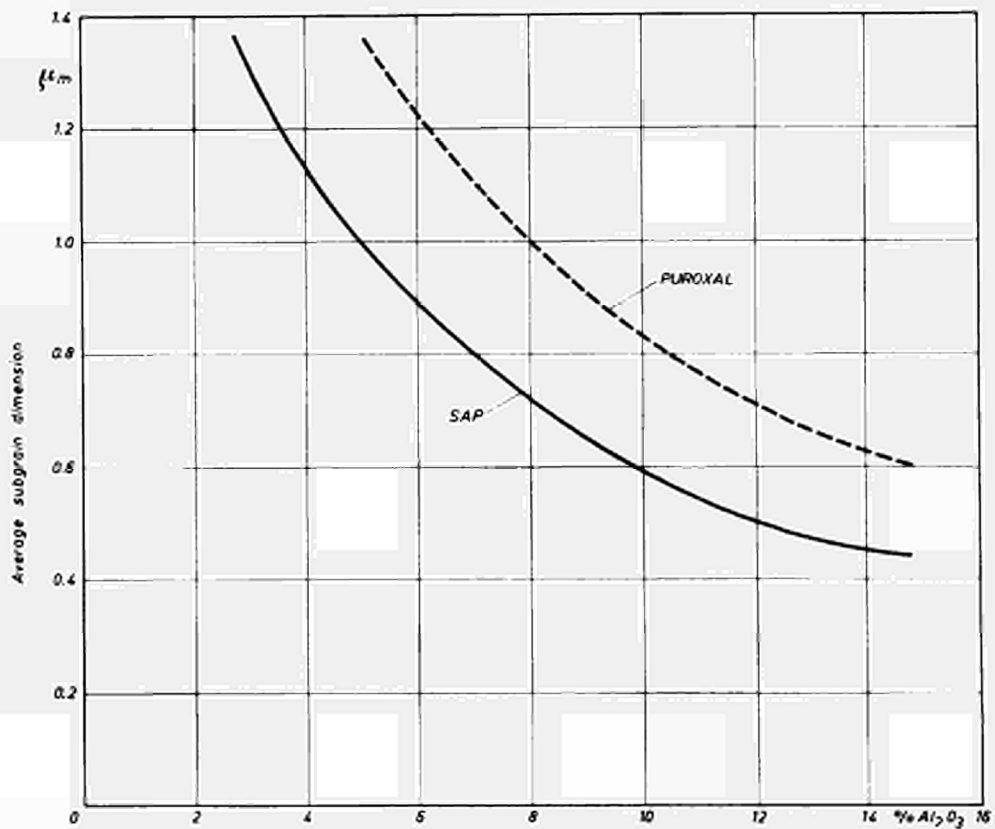


Fig. 15 - Average Grain size vs. Al_2O_3 content. (Plate N. 31164/6).

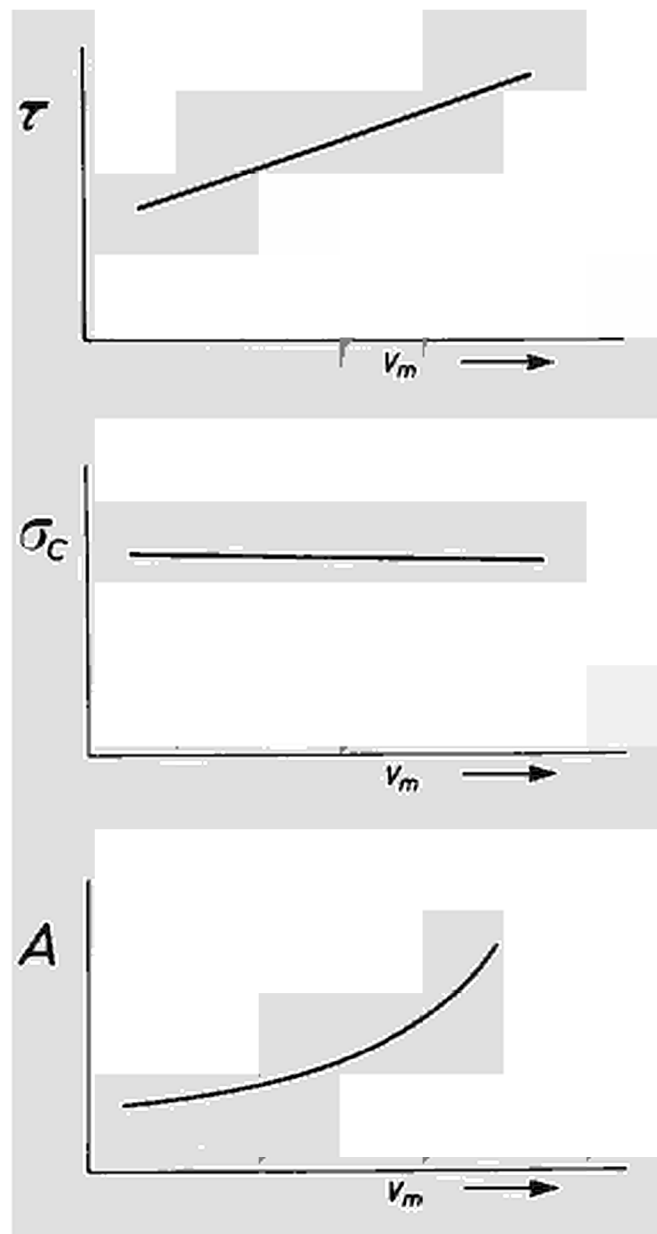


Fig. 16 - Dependence of the rate of strain (v_m) on elongation as a function of τ and σ_c . C^* is a constant.

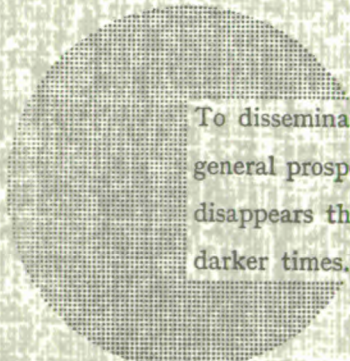
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Alfred Nobel

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