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# Formation of Aluminium Pearls on the Surface of SAP Alloys

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An abnormal phenomenon of aluminium "pearl"—formation at the surface of Al–$Al_2O_3$ alloys was observed occasionally in quite different experimental conditions. In order to obtain more information, a series of hot-hardness and hot-compression tests was performed on SAP specimens. Qualitative correlations between the experimental variables and the experimental results are described; tentative explanations are related to Sherby's hypothesis of a continuous alumina network and to the presence of a non-oxidising atmosphere.

## 1. Introduction

An anomalous phenomenon was sometimes observed at the surface of SAP (Al–$Al_2O_3$) alloys: occasionally, in quite different experimental conditions, small quantities of aluminium, normally like shining pearls, were found at the surface of the specimens.

Typical experiments which revealed this phenomenon are the following:

(a) When SAP was heated in the vacuum-furnace of a hot-stage microscope, the pearls appeared sometimes at temperatures of about 640° C, about 20° C below the melting point of the aluminium matrix. For instance, SAP with a nominal content of 14 wt % $Al_2O_3$ heated in the hot-stage microscope at the temperature of 620° C did not reveal any change in the structure after 1 h at this temperature. When the temperature was raised to 640° C, pearls appeared as shown in fig. 1 [1].

(b) Thermal treatments at temperatures from 600 to 640° C, for 2000 h were made on SAP cylindrical specimens enclosed in argon-filled quartz capsules. After the treatment, some specimens had the surface covered by several small shining pearls [2]. Appearance and consistency of these pearls were those of pure aluminium; X-ray Debye-Scherrer patterns confirmed that the composition was aluminium. The same phenomenon was observed after thermal treatment at 600° C for 1000 h and at 500° C for 4000 h [3].

(c) A study of the high-temperature hardness of SAP alloys [4] included tests at 450° C for times up to 50 h. For the longer periods a considerable number of small protuberances was observed around the indentations.

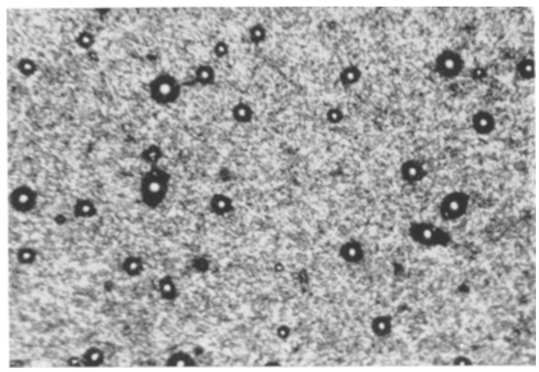

*Figure 1* SAP/14 wt. % $Al_2O_3$, observed in the hot-stage microscope, at 640° C (× 170).

## 2. Experimental

In order to give an interpretation of these phenomena a series of hardness, metallographic and hot compression tests were carried out on SAP specimens.

### 2.1.

Brinell hardness tests were made on SAP containing 7 wt % $Al_2O_3$ at 450° C for periods longer than 15 h. Near the indentations pearls are visible, as shown in fig. 2. Sections of the specimens were examined in order to enable micrographic observation of the pearls: an

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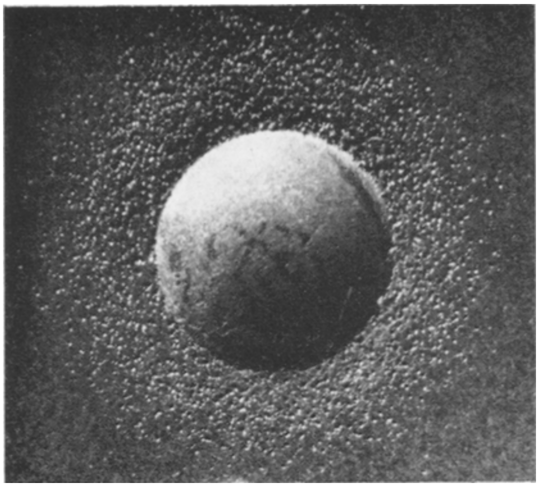


*Figure 2* Indentation in SAP/7 wt % $Al_2O_3$: Brinell hardness test at 450° C for 15 h (× 17).

example is shown in fig. 3. The following conclusions can be drawn:

(a) The small protuberances (pearls) are almost completely oxide-free.

(b) The protuberances correspond to zones, within the SAP, with low oxide content, zones which are present in the SAP alloys as inhomogeneities: the "pearls" are always connected with these "white" zones (fig. 3).

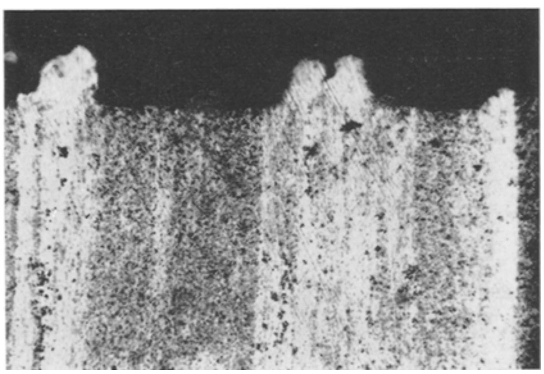


*Figure 3* A micrographic section through some of the pearls shown in fig. 2 (× 420).

2.2.

Compression tests were carried out on cubic specimens of SAP alloys with different oxide contents (4, 7 and 14 wt %). Attention was paid to the extrusion direction: "white" zones, which correspond to almost oxide-free aluminium, are well aligned in this direction.

*2.2.1.*

A first series of compression tests was made with the compression direction perpendicular to the extrusion direction. The tests were performed at different temperatures and stresses and for different periods of time, ranging as follows: temperature: 400 to 620° C; time: 1 to 90 h; stress: near (above or below) the yield point of the material at the temperature of the test.

In a certain number of tests it was possible to observe the "pearls" which always appeared in the extrusion direction, i.e. on the faces perpendicular to the extrusion direction. The results of the observations however were not such as to permit a quantitative analysis of the phenomenon. This was due mainly to two factors:

(a) Non-uniform dispersion of the pearls over the surface of the test specimens, caused by the rather complicated stress distributions in the compression tests.

(b) Impossibility of establishing a consistent correlation of the pearls (number or volume) with the variable experimental parameters.

Nevertheless some general remarks can be made:

The micrographic observation confirms that the pearls are preferably found at the end of the "white", aluminium-rich zones (fig. 4).

The pearls are more easily generated in SAP containing 14 wt % $Al_2O_3$ than in SAP with lower oxide content.

The pearls were observed only on the specimens compressed at temperatures between 450 and 500° C. Above 500° C the specimens are

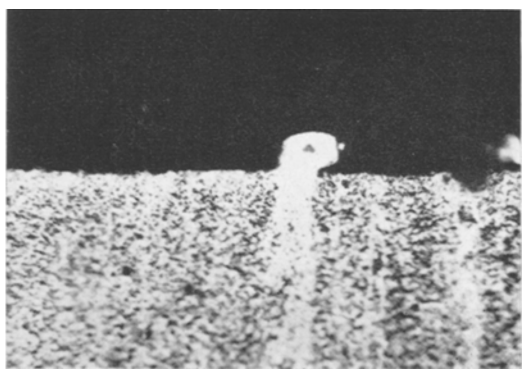


*Figure 4* Aluminium "pearls" at the surface of a SAP/14 wt % $Al_2O_3$ specimen after a compression test (× 420).

highly deformed and this fact may mask the pearl formation.

Number and size of the pearls increase with time, at constant temperature. For example, at 500° C the first pearls appear already after about an hour and they increase in number and volume for longer annealing times. After about 60 h incipient saturation is observed.

The pearls are observed in compression tests only at stresses near the yield point, above or below. Stresses much higher than this value will lead to rupture in a time not sufficient for the formation of the pearls. Below the yield point, a decrease of the stress results in an increase of the time necessary for observing the pearls. The above mentioned observations show that for very long periods pearls may appear without external pressure.

The formation of pearls is more pronounced in distorted zones of the surface. High concentrations of pearls were observed in scratches previously made at the surface of a SAP 4 wt % $Al_2O_3$ specimen, after 16 h at 500° C.

*2.2.2.*

A second series of compression tests was performed with the compression direction parallel to the extrusion direction: in this experiment the lower plate is provided with a hole, as indicated in fig. 5. With a SAP 14 wt % $Al_2O_3$ specimen, at the temperature of 500° C, pearls appear at the edge of the hole (see fig. 5) after about an hour. The observed phenomena are about the same as in the experiments described previously (section 2.2.1), as shown in fig. 6, and also in this case a more quantitative approach to the problem is particularly difficult because of the non-uniform and not well reproducible formation of the pearls.

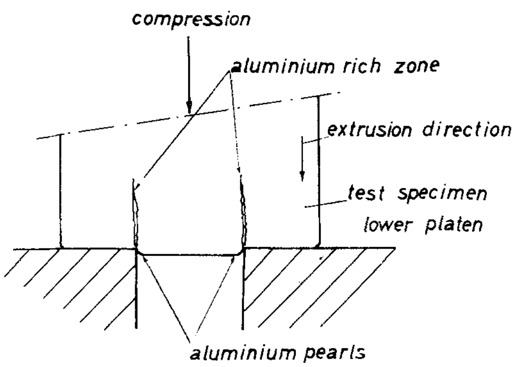



*Figure 5* Compression test with extrusion direction parallel to the compression direction.

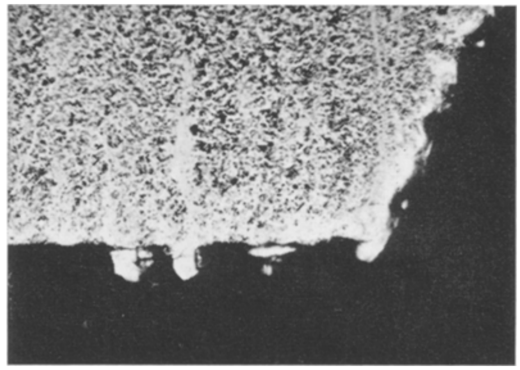


*Figure 6* Pearls observed in SAP/14 wt % $Al_2O_3$ specimen after the compression test schematised in fig. 5, 500° C, 16 h (× 420).

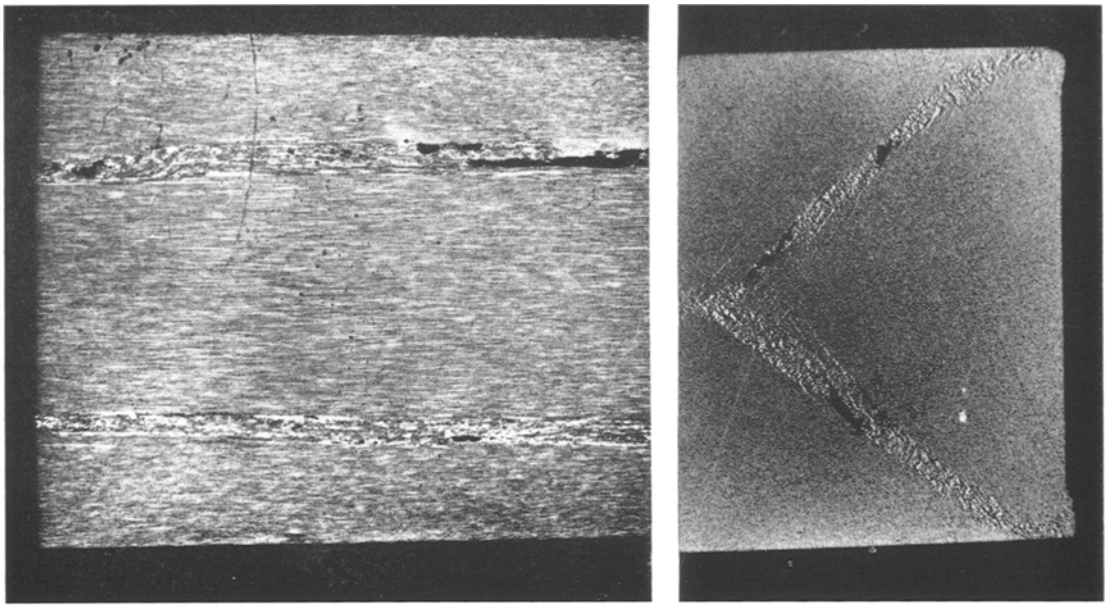
2.3.

Another unusual phenomenon was observed in the SAP specimens after the compression tests executed in a direction normal to the extrusion direction. Segregation of aluminium from aluminium oxide induced the formation of zones of pure aluminium in preferred planes (fig. 7). These planes, starting at the corners of the specimens, are the planes of the main deformation in cubic specimens under compression [5, 6].

The same phenomenon of segregation with formation of aluminium-rich zones is observed in the specimens after the compression test schematised in fig. 5: the plane of maximum shear stress are characterised by "white" zones (fig. 8).

## 3. Discussion

Only quantitative correlations between the different factors which affect the described phenomena could lead to an exact interpretation of pearl-formation. The lack of such data permits only tentative explanations. The main fact, which is common to all observations, is that the pearls appear in general on the surfaces normal to the extrusion direction, and any attempt at an explanation should start from this point. The oxide plates in the material are oriented parallel to the extrusion-direction, and plastic flow of aluminium certainly encounters less resistance in this direction than in a direction normal to it.



(a) (b)

*Figure 7* Sections of a SAP/14 wt% $Al_2O_3$ specimen after a compression test at 500° C for 16 h. Extrusion direction: (a) from left to right (b) normal to figure plane. Compression direction: vertical (× 4.5).

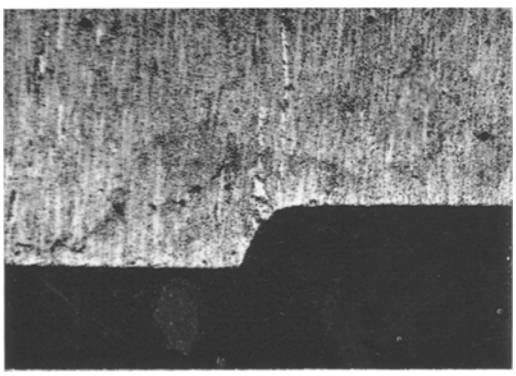


*Figure 8* Aluminium-rich zones in a SAP specimen after the compression test of fig. 5 (× 55).

Furthermore, there is not very much adherence between the oxide and the matrix and so the metal/oxide interfaces, lying mostly parallel to the extrusion direction, may be zones of easy gliding.

But these circumstances alone would not explain the protrusion of separated pearls. There must be a resistance which holds back the mass of the material, allowing an exit only at certain points. This resistance may be supplied by a more or less continuous alumina network, as supposed by Sherby *et al.* [7-10] or else by a skin on the surface which is generally quite resistant but shows weak points where the quasi-liquid metal comes out. There is some experimental evidence for both cases.

It is interesting that there seems to be a distinct relation between zones of shear and the appearance of the pearls. Zones of shear furthermore become depleted of alumina. In the Sherby model, shear stresses at the particle-matrix interfaces may result in a separation of aluminium between the cells elongated in the extrusion direction, allowing the metal to move; this is a kind of micro-extrusion in the elongated channels and results in pearl-formation at the surface, in areas poor in alumina. A scheme of the proposed structure is represented in fig. 9. This model implies pearl-growth in correspondence to the oxide-free zones and appearance of the pearls preferably at the surfaces perpendicular to the extrusion direction, which are experimentally confirmed.

This scheme is also consistent with the fact that the number of pearls in the case of SAP/14 wt % $Al_2O_3$ is greater than in alloys containing less oxygen: with the higher oxide content, the structure is nearer to the scheme of Sherby's hypothesis and, according to the proposed

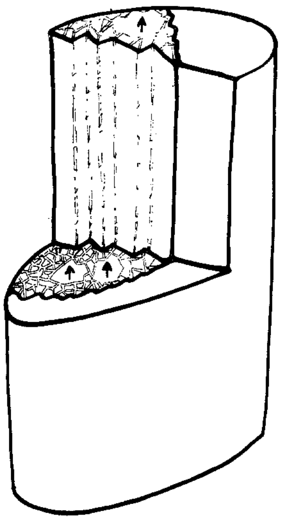


*Figure 9* Scheme of the elongated cell structure in an extruded SAP type alloy (not to scale) showing alumina walls separating region of aluminium matrix. Arrows show direction of the aluminium movement in the oxide-free zones ("microextrusion channels").

mechanism, pearl-formation is more probable than in the alloys with an incomplete network.

Pearls have been found, without application of pressure, only under vacuum or in an oxygen-free atmosphere, where the surface oxide layer remains relatively thin. The formation of such an oxide layer may on the other hand be disturbed by the presence of the dispersed oxide as is shown by a certain difficulty in obtaining anodic oxide layers on SAP. The intersection of the oxide/matrix interface with the surface may provoke weak points in the oxide, and zones where this oxide is furthermore damaged by a scratch or by stretching during shear are even more vulnerable and prone to pore formation.

Distinction between the relative importance of the two models is not possible on the basis of the existing experimental results. It might be possible to obtain more evidence by doing other tests, for instance, tests in a strictly oxygen-free atmosphere, avoiding the formation of surface oxide by means of a careful preparation by ion-etching.

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