

English Translation of Investigations on Long Term Behaviour of Lead and Lead-Alloys
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1. Introduction

The creep behaviour of technical materials is at present being intensively investigated, particularly since its significance has been identified not only for cases of constant loading but also for instances of vibrating stress (1). An awareness of the creep behaviour based on long-term experiments, such as have been carried out systematically for decades on heat-resistant materials (2) and for some years on non-ferrous metals, (3) still remains an essential precondition for the dimensioning of components and for an estimation of their service life under given operational conditions. Even if strenuous efforts are being made to come to terms increasingly with the processes which take place during creep, and their causes, and not simply to be satisfied with the recording of expansion-time curves under constant stress, the physical laws which have been explained hitherto are still not sufficient to be able to forecast the creep behaviour of a material with a satisfactory degree of certainty from short-term experiments. The substantial effort and expenditure in terms of technical resources and time involved in creep tests over many thousands of hours (with steels, up to 200,000 hours) is something that for the foreseeable future cannot be avoided, if we want to provide the designer with reliable data about the long-term behaviour of the materials.

With lead, and a number of lead materials, as a result of the low recrystallisation temperature and low strength already inherent at room temperature, creep leads to deformations which may no longer be tolerable for a structural design, in a similar way to that in which steels are observed when they reach substantially high temperatures. Reference has already been made in the standard works (4, 5) about lead and lead alloys to the time and temperature dependency of the strength properties of lead materials. Most recently, reports have been forthcoming from experiments over a period of about 10 years conducted on the use in the chemical industry of a number of selected alloys (6, 7). Arranged by a joint programme relating to the creep behaviour of non-ferrous metals (copper, nickel, aluminium, titanium, and alloys), which have been running since 1962 at the *Dechema Technical Commission on "Materials and Design in Chemical Apparatus Construction"*, the lead production and processing industry has proposed the systematic investigation of 22 lead materials. The impetus for this came from a decision by the *Bleiberatung e.V.* lead consultation association in 1965, from which the desire for investigations of this nature was presented to the *DECHEMA*. As test facilities for these long-term experiments, in a working group from the *Dechema Technical Commission* referred to, the undertaking involved the companies of *Bayer AG, Leverkusen, Bleiwerk Goslar KG, Goslar, Hoechst AG, Frankfurt-Höchst, Aug. Schnakenberg u. Co., Wuppertal, and Vereinigte Zinkwerke GmbH, Stolberg (Rhld.)*. Supplementary investigations were provided by the companies of *Bleiwerk Goslar KG, Goslar, Aug. Schnakenberg u. Co., and Vereinigte Zinkwerke GmbH*.

2. Materials examined

When selecting the materials to be examined, the basis taken was the classification according to DIN 1719 and DIN 17 641. The principle was adopted in this situation that in the first instance the main alloy constituents copper, antimony, and arsenic would be determinant. At the same time the influence of bismuth admixtures was to be investigated, since for reasons of economy the bismuth contained in the ores is occasionally not removed when smelting the lead. Further alloy additives are introduced for use in the chemical industry, predominantly to increase the corrosion resistance. As well as copper, this involves in particular additives of nickel, tin, silver, and more recently palladium. Their effect consists of an influence on the microstructure formation, whereby a more or less pronounced inclination to passivation in the event of sulphuric acid corrosion is attained due to the formation of a sulphate cover layer. The corrosion-inhibiting effect depends heavily on the combination of these additives and the determination of their quantity. It is not always the case that increasing the corrosion resistance also improves the creep behaviour. Of the extensive literature relating to alloy technology, mention may be made here with regard to corrosion resistance, as well as (5), only to (8-10). The latter set also discusses the simultaneous increase in creep resistance of corrosion-resistant alloys, such as has been determined, for example, for the four-material alloy PbCuSnPd.

For the research programme, in the first instance alloy additives of this type were missed out, and, as well as refined lead 99.99, only the two-substance and three-substance alloys with the components copper, antimony, and arsenic (copper refined lead and hard lead) were chosen, in different compositions. These materials, the abbreviated test symbol used in the study, their product form, and their chemical composition are summarised in *Table 1*. *Table 2* shows the tensile strength and the 0.2% and 1% tensile yield strength at room temperature. A distinction therefore must be drawn between the five groups of materials:

Refined lead Pb 99.99

Copper refined lead Pb 99.9 Cu

Hard lead with 1% by mass antimony

Hard lead with 4% by mass antimony

Hard lead with 8% by mass antimony

In the case of refined lead and copper refined lead, one low-bismuth and one bismuth-rich melt were examined in each case. Of the hard lead with 1% by mass antimony, one arsenic-free melt and one containing arsenic, both low-bismuth, were tested, as well as a bismuth-rich melt containing arsenic. For the hard lead with 4% and 8% by mass of antimony, one melt each without arsenic and one containing arsenic were included in the test.

For the four substance groups of refined lead, copper refined lead, and hard lead with 1% or 4% by mass of antimony, the material examined in each melt took the form of rolled sheet 10 mm thick and pressed round bars with 10 mm diameter. The two melts of hard lead with 8% by mass of antimony were tested in the product form of pressed round bars with 10 mm diameter and cast cylindrical bodies with 10 mm diameter.

The microstructures of the materials in the initial state are reproduced in *Figures 1 to 6* as transverse sections.

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Table 1. Materials, abbreviated test symbols, product form, and chemical composition of the materials examined

Table 2. Tensile strength and 0.2% and 1% tensile yield strength at room temperature (extension rate: 5 mm/min)

Table 3: Limiting creep stresses and creep resistance of refined lead 99.99

F 1 + F 3 rolled sheet, F 2 + F 4 extruded; F 1 + F 2 low-bismuth, F 3 + F 4 rich in bismuth

Table 4: Limiting creep stresses and creep resistance of refined lead copper (Pb 99.99 + 0.04 – 0.05% Cu)

C 1 + C 3 rolled sheet, C 2 + C 4 extruded; C 1 + C 2 low-bismuth, C 3 + C 4 rich in bismuth

Table 5: Limiting creep stress and creep resistance of hard lead with 1% antimony

H 1, H 2, H 5 rolled sheet, H 2, H 4, H 6 extruded; H 1 + H 2 low-arsenic and low-bismuth, H 3 + H 4 containing arsenic and low-bismuth, H 5 and H 6 containing arsenic and rich in bismuth

Table 6: Limiting creep stress and creep resistance of hard lead with 4% antimony

S 1 + S 3 rolled sheet, S 2 + S 4 extruded; S 1 + S 2 arsenic-free, S 3 + S 4 containing arsenic

Table 7: Limiting creep stress and creep resistance of hard lead with 8% antimony

W 1 + W 3 rolled sheet, W 2 + W 4 cast; W 1 + W 2 arsenic-free, W 3 + W 4 containing arsenic