## SHORT REPORTS AND LETTERS TO THE EDITOR INFLUENCE OF PRESSURE ON THE MUTUAL DIFFUSION OF METALS STUDIED IN CONNEXION WITH THE APPEARANCE OF DIFFUSION POROSITY\* Ya.Ye. GEGUZIN and A.S. DZYUBA

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1. The high-temperature annealing of crystalline solids in which there are excess vacancies or a vacancy "source" is known to lead to the formation of what is known as diffusion porosity [1]. In [2] it was found experimentally that external volumetric compression does affect the process of the nucleation and development of pores of diffusion origin. Moreover it was found that comparatively small pressures, of the order of  $\sigma/L$  ( $\sigma$  is the specific surface energy of the crystalline solid and L is the mean linear dimension of nucleating microcracks), will prevent the formation of pores, while at  $P \sim 10-10^2 \text{ kg/cm}^2$ , which corresponds to the reasonable value  $L \sim 10^{-4} - 10^{-5}$  cm, practically no diffusion porosity will arise. This has been confirmed experimentally in works where the mutual diffusion [3] and processes of the high-temperature annealing of defects has been studied in single crystals [4, 5].

The present work was undertaken with the aim of obtaining further information on this effect, particularly that which would emerge from a comparison of metallographic data and data on the microhardness distribution in the diffusion zone of specimens.

2. In the experiments described below single-phase brass type L-70 and technically pure copper were used. The diffusion equalisation of the zinc concentration in a brass-copper system is known to be accompanied by the development of diffusion porosity in the brass (see [1]).

The specimens were prepared as follows. Into a copper tube which had been carefully annealed and etched in nitric acid a brass wire was inserted after it had first been freed of various kinds of surface inclusions and oxides by mechanical polishing and etching in a solution of ammonium persulphate in ammonia. A really reliable and sensitive contact

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between the brass and copper was achieved by drawing the copper tube with the brass core through a set of wire dies. The wire produced in this way was cut up into specimens 10 mm long and 1.5 mm in diameter.

The specimens were given a diffusion annealing in an autoclave with a volume of 20 cm<sup>3</sup> in an argon atmosphere 99.99 per cent pure. The autoclave was designed [6] so that the pressure in the working chamber could be varied from 1 to 100 atm. The temperature was adjusted and maintained by a two-position electro-mechanical heat regulator with an accuracy of  $\pm 2^{\circ}$ C. A chromel-alumel thermocouple was used as the temperature pickup.

The experiments consisted in the following. Specimens produced as described above were given an isothermal anneal at various different pressures. The anneal was carried out at 800° and pressures of 1, 10, 25, 50 and 100 atm. At each pressure the annealing time was varied from 0.5 to 19 hr. After each anneal a metallographic section was produced in the plane perpendicular to the axis of the specimen and the microstructure was studied. Fig. 1 shows typical structures of specimens which had been annealed for 6 hr at various different pressures. This collection of photographs provides qualitative confirmation of the effect of volumetric compression on the nucleation and development of pores which have arisen in the process of mutual diffusion. As with the aerlier experiments [2], a pressure of  $p \simeq 10$  atm reduces considerably the development of the diffusion porosity. The slight quantitative differences in the effect of pressure in these experiments and those described in [2] may be due to the different purity of the brass. This would lead to different kinds of micro-imperfections which would become condensation centres for excess vacancies. If the linear dimension of this kind of imperfection is assessed by means of the obvious relation  $r \simeq \sigma / p^*$  (p\* is the minimum

pressure required for the deactivation of an imperfection), at  $\sigma \simeq 10^3 \text{ ergs/cm}^2$  and  $p^* \simeq 100 \text{ kg/cm}^2$ , one gets the reasonable value  $r \sim 10^{-5} \text{ cm}$ .

3. We also obtained data on the microhardness distribution. In a general case a change in microhardness in the region of the brass core in the course of a diffusion anneal may be due to two reasons, variation in the sinc concentration and the appearance of porosity. no shown by control tests, the first of these reasons hardly appears under these annealing conditions. This is demonstrated in particular by the fact that the microhardness in conditions where porosity is impossible due to pressure ( $p \simeq 100$  atm), hardly changes at all with time although there is partial loss of the zinc (Fig. 2). This provides basis for the assumption that the change observed in the microhardness must be due to the presence of visible and, within the resolution range of the metallographic method, invisible pores.

Figure 3 gives the characteristics for the microhardness distribution along the radius of cylindrical specimens which had been annealed under a pressure of 1 and 100 atm. The maximum on curve 2 is due to the fact that porosity arises in the latter part of the annealing, not only in the brass itself, but also in the regions of copper adjacent to it. Here it must be emphasized that a reduction in microhardness is not only observed around visible pores, but also in regions which were found to be metallographically free of pores. This can be understood if it is assumed that diffusion porosity is also formed beyond the region where loss of zinc was detected. This conclusion is consistent with the earlier established fact [1] according to which the saturation of vacancies, which is decisive for the development of diffusion porosity, is of the order of magnitude  $\Delta \xi / \xi \sim 10^{-2}$ . This also agrees with data on the nucleation of diffusion pores, which was obtained in experiments with the low-angle scattering of X-rays [7].

The experiments described have thus not only confirmed that low pressures do influence the nucleation and development kinetics of diffusion porosity, in the form of exceedingly disperse pores, which causes a reduction in microhardness, does develop in regions where it cannot be detected metallographically.

Translated by V. Alford.



FIG. 1. Structure of specimens after annealing at 800°C for 6 hr and at the following pressures:

 $a = 1; b = 10; c = 25; d = 50; e = 100 \text{ kg/cm}^2.$ 

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annealing in an he autoure in the to 100 atm. ained by a ulator mel re pickup. wing. were given pressures. ressures ssure the hr. After s produced the speci-Fig. 1 hich had t pressures. Jualitative ompression es which ision. As re of opment titative ese ay be due vould ons for of this s of the um



FIG. 2. Microhardness of the brass core of a specimen as a function of annealing time at 800°C and different pressures:



- 2 = annealing for 1 hr at 100 kg/cm<sup>2</sup> followed by
- annealing at 1 kg/cm<sup>2</sup>;





FIG. 3. Distribution of microhardness along the radius of cylindrical specimens after annealing at 800°C and pressures of:

1 - 100;2 - 1 kg/cm<sup>2</sup>.

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