THE EFFECT OF HIGH TEMPERATURE ANNEALING IN DIFFERENT ATMOSPHERES ON PORE FORMATION IN SURFACE REGIONS OF ODS MATERIALS

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Resume
Mechanically alloyed oxide dispersion strengthened materials MA 6000 and MA 956 have been annealed at 1100°C for 24 and 240 hours in air, vacuum of $10^{-3}$ Pa and hydrogen. Mechanical properties after annealing were then tested at room temperature. Mass changes due to oxidation and evaporation were measured. Fracture surfaces, microstructure of the materials and changes in chemical composition were examined. The results of mechanical tests yielded generally only small changes of mechanical properties after all expositions. On fracture surfaces and polished sections, formation of voids and altered microstructure sub-surface zones in both materials has been studied. Different mechanisms causing void formation are discussed and proposed. Also the mechanisms and reaction sequences of surface oxidation reactions are examined.

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1. Introduction
The use of Oxide Dispersion Strengthened (ODS) materials in high temperature applications is widely considered as very promising step towards achieving higher temperatures and thus higher efficiency values in cases such as gas turbine, in prospective fusion reactors or other most demanding high temperature applications. These alloys combine two important characteristics - good long term stability of microstructure providing creep strength and also high temperature corrosion resistance through surface passivation layers [1, 2, 4, 5].

In the case of the two materials under examination in this contribution - MA956 and MA 6000, iron and nickel form the base of the materials matrix respectively. Strengthening yttrium oxide particles are added to the microstructure by means of powder metallurgy route in both cases. The oxide layers that formed during high temperature exposition represent a basic and essential mechanism by which a material can protect itself from aggressive environment. In the presented experiments, the materials were annealed in vacuum, air and hydrogen all at the temperature of 1100 °C.

Different mechanisms may be involved in the formation of pores in superalloys during annealing [3, 8 - 11]. These may be related to the reaction of the material with the surrounding environment, the reaction to an external stress applied on the sample under investigation, or the technology of producing a particular material.

The first group of mechanisms explains the creation of cavities by condensation...
of vacancies in the metal matrix of the material [3, 6 - 8]. During oxidation, migration of metal ions through the oxide towards the free surface is compensated by the flow of vacancies into the material through the oxide. These vacancies should normally disappear at the oxide-metal interface. When this annihilation does not occur, the vacancies pass into the bulk metal substrate, where again there are possibilities for their extinction at grain boundaries or at interface of the matrix and noncoherent particles. If these mechanisms do not appear to be sufficient, condensation of vacancies must occur. This vacancies condensation is then a mechanism of formation of cavities in areas close to the surface. Another mechanism that may be included in this group lies in dissolving some of the phases in the bulk of the material. Should the formation reactions of oxides on the surface require an element that forms a carbide phase in the material bulk for example. A cavity copying the original shape and size of the dissolved particle is left in the material in such case. The flow of atoms from the disintegrating particle towards the surface is to some extent compensated also by flow of vacancies in the volume of the dissolved particle.

The second group includes mechanisms of formation of cavities involving mechanical stress in the material [3, 6 - 8]. The cavities in the ferritic ODS materials produced by powder metallurgy are attributed to the relaxation of the internal stresses produced by the pressing or extrusion of powder materials. This approach assumes that due to the different properties of the matrix and the oxide reinforcing particles, local stresses builds up in the material, which are then relaxed by diffusion of the matrix atoms and subsequent formation of the cavities (at a sufficient temperature).

The third group of mechanisms relates to the formation of cavities by gas molecules accumulation in the material [3, 6 - 8]. Especially for nickel-based materials, the formation of cavities filled with CO2 / CO mixtures is mentioned in the literature. The formation of such cavities is related to the reaction of the carbon dissolved in the metal matrix of the material and the oxygen atoms that diffuse into the material from the external environment. Preferably, at the grain boundaries, new pores can be formed. These are not related to the formation of an oxide layer on the surface of the material.

In connection with materials produced by powder metallurgy, the formation of cavities filled with gas used as a protective atmosphere during powder milling is considered. The molecules of this gas (most commonly argon) are captured in individual powder particles; exposure to high temperatures leads to the release of these molecules and their coupling in the cavity.

Identification of the porosity thus produced may be facilitated by the assumption that the pores should be uniformly present throughout the volume of the material [7, 12, 13]. There is no clear consensus about the mechanism of formation of pores during high temperature exposure ferritic ODS materials. In addition to the mechanism involving the gas molecules trapped in the powder during its production, the possibility is also mentioned that the formation of cavities in the material allows the relaxation of internal stresses introduced into the material during deformation manufacturing (e.g. extrusion). The presented work discusses the viable pore formation mechanisms based on chemical changes and morphology features of the observed pores.

2. Experimental setup

ODS superalloy materials MA 956 and MA 6000 were used in the tests both materials were manufactured by Special Metals Co. purchased through BIBUS Company. Nominal chemical composition of these materials is presented in Table 1.
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### Table 1

<table>
<thead>
<tr>
<th></th>
<th>wt. %</th>
<th>Cr</th>
<th>Ni</th>
<th>Fe</th>
<th>Al</th>
<th>Mo</th>
<th>W</th>
<th>Ta</th>
<th>Ti</th>
<th>Y2O3</th>
<th>Zr</th>
<th>C</th>
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<tr>
<td><strong>MA 6000</strong></td>
<td></td>
<td>14.90</td>
<td>bal</td>
<td>0.78</td>
<td>4.46</td>
<td>1.97</td>
<td>3.93</td>
<td>2.03</td>
<td>2.60</td>
<td>1.09</td>
<td>0.16</td>
<td>0.03</td>
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<tr>
<td><strong>MA 956</strong></td>
<td></td>
<td>19.00</td>
<td>0.10</td>
<td>bal</td>
<td>4.39</td>
<td></td>
<td></td>
<td></td>
<td>0.38</td>
<td>0.52</td>
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<td>0.03</td>
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### Table 2

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<tr>
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<th>AIR 1100°C</th>
<th>VAC 1100°C</th>
<th>HYD 1100°C</th>
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<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>g/mm² 24</td>
<td></td>
<td>-3.94·10⁻³</td>
<td>-8.24·10⁻⁶</td>
<td>1.93·10⁻⁶</td>
</tr>
<tr>
<td>g/mm² 240</td>
<td></td>
<td>-5.22·10⁻³</td>
<td>-5.00·10⁻⁵</td>
<td>2.89·10⁻⁶</td>
</tr>
<tr>
<td><strong>MA956</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>g/mm² 24</td>
<td></td>
<td>4.49·10⁻⁶</td>
<td>-4.40·10⁻⁵</td>
<td>1.77·10⁻⁶</td>
</tr>
<tr>
<td>g/mm² 240</td>
<td></td>
<td>9.41·10⁻⁶</td>
<td>-3.25·10⁻⁴</td>
<td>3.95·10⁻⁶</td>
</tr>
<tr>
<td>g/mm² 1000</td>
<td></td>
<td>1.84·10⁻⁵</td>
<td></td>
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</tr>
</tbody>
</table>

For evaluating the mass changes during annealing, specimens of 10×10×3 mm were cut and polished on SiC paper 2000 grit. These specimens were also used for microstructural examination of the materials.

All specimens were annealed at 1100 °C for 24 and 240 hours in air, vacuum of 10⁻³ Pa and hydrogen of normal pressure. Ma 965 samples were annealed also for 1000 hours. Non-annealed specimens were also examined for comparison means. Commercially available hydrogen of 99.99% purity was used in the experiment without any further treatment.

After annealing, the samples were cut to half and metallographic samples were prepared by standard technique. Light and electron microscopy was used to characterize the microstructure and EDS was used for local chemical analysis. In this paper, particularly the results obtained by Philips XL30 SEM coupled with EDAX EDS analyser are shown. The weight changes were measured by analytical Sartorius scale.

3. Results of measurements

3.1 Mass changes

All samples were weighted before and after the annealing. The results are contained in Table 2.

Annealing in air caused weight loss on MA6000 samples and weight gain on MA956 samples. While MA6000 grows oxide layers that fall off the samples, MA956 grows a homogeneous oxide layer that adheres to the surface and adds to the total weight of the sample. After vacuum annealing, both samples showed mass loss. In both cases this is due to evaporation, mainly of chromium from the matrix of both materials. The evaporation of bulk matrix could not be ruled out either. In MA956 it appears so that part of the matrix evaporated, leaving only fine, yttrium, aluminum and titanium containing particles on the surface. Annealing in hydrogen atmosphere produced mass gain on both materials. Both materials developed well adhering oxide layers in this case.

3.2 Microscopic analysis

3.2.1 Air annealing MA6000

When the MA6000 is annealed in the air, a composite layer of aluminum and chromium oxides with embedded nickel matrix particles is formed on the surface (Fig. 1, Fig. 2). The thickness of these layers can be determined only approximately as the oxidation surface attack is uneven and locally falls off. After 24 hours of annealing at an annealing
temperature of 1100 °C, the oxide layer has a thickness of 25 μm, after 240 hours there is already a significant loss of oxidation products, the maximum thickness of oxide layer exceeds 50 μm. The uppermost layer of this system consisted of chromium oxide, particles of the original matrix were sealed in the oxide layer. After 240 hours of annealing, the majority of the oxide layer has fell off from the surface of the sample, its composition can only be observed on residues in deeper affected areas. This residual layer is formed by aluminum oxide and the now also oxidized nickel matrix particles.

During annealing in the air, pores gradually formed beneath the surface of the material as far as 100 μm after 24 hours, and 300 μm after 240 hours. After 24 hours (Fig. 1), pores are formed by separating individual particles from the surrounding matrix. After 240 hours (Fig. 2), hollow pores are already visible. Particles of high titanium, aluminum and yttrium content (Fig. 3) are found on the inner surface of the pores together with residues of the nickel matrix with increased titanium content up to 20 wt. % and low chromium.

Slight decrease in chromium content in matrix of the material has been measured in the vicinity of the surface. Correspondingly to the observed mixed oxide layer with the matrix residues on surface, increase of aluminum and oxygen content was measured on surface.

3.2.2 Air annealing of MA956

With annealing in the air, a compact layer of Al₂O₃ oxide containing small particles with high titanium, aluminum and yttrium content was formed on the MA956 samples. The thickness of this layer is 2 μm after annealing for 24 hours, 6 μm for 240 hours, and 10 μm for 1000 hours.
of annealing. The oxide layer adheres well to the surface even after tensile tests (Fig. 4). EDS analysis of the chemical profile of the samples showed oxygen and aluminum in the surface oxide layer; below this layer there was no measurable change in chemical composition compared to the original state.

Newly formed pores inside the material in the structure of MA956 samples were found rather in the core part of the sample than near the surface. These pores have a characteristic oval shape stretched in the rolling direction of the original material. They are also visible on the fracture surfaces, the inner surface of these pores is covered with small particles that have a high content of yttrium, titanium and aluminum (Fig. 5).

3.2.3 Vacuum annealing of MA6000

Vacuum annealing of MA6000 samples created pores open to the surface. The pore area reaches 20 μm below sample surface after annealing for 24 hours and 250 μm after annealing for 240 hours (Fig. 6). The pores contain small particles with high content of aluminum, titanium and yttrium on the inner walls or residues of the matrix material. In the diffusion affected subsurface area of the sample also particles of mixed tungsten and molybdenum carbides were found. On fracture surface of the annealed sample a recrystallized 8 μm wide area is visible under the surface of 8 μm (Fig. 7). This area corresponds to higher titanium and lower chromium content. Chemical analysis revealed a chromium depletion towards the surface of the material and a slight increase in titanium content. The recrystallized area at the fracture area corresponds to the lowest measured chromium concentration.
3.2.4 Vacuum annealing of MA956

During vacuum annealing, the matrix evaporated to certain extent, which resulted in thermally etched surface with a high concentration of (oxide) particles of yttrium, aluminum and titanium (Fig. 8). The chromium and iron content decreased towards surface and due to the accumulation of minor particles on the surface and in its vicinity there is an increase in yttrium and aluminum concentration.

Similarly to air annealing samples, porosity was formed in the material (Fig. 9). The pores are concentrated more in the inner bulk of the sample than in the subsurface areas. The pores have internal surfaces covered with small particles of high content of yttrium, aluminum and titanium.

3.2.5 Hydrogen annealing of MA6000

During annealing in hydrogen, a very thin layer of aluminum oxide was formed on the MA6000 surface, through which particles with high titanium and yttrium (Fig. 10, Fig. 11) penetrated. This layer has a thickness of 1 μm after annealing for 24 hours and 2 μm for 240 hours. Chemical analysis showed a decrease in chromium content immediately below the oxide layer formed by Al₂O₃. The profile also shows an increase in titanium, tungsten and yttrium content in the oxide layer.

3.2.6 Hydrogen annealing of MA956

A continuous aluminum oxide layer was formed on the surface of the samples after annealing in hydrogen, containing fine particles with high content of yttrium and titanium (Fig. 12, Fig. 13). These particles penetrate the entire oxide layer and some of them appear on the surface (Fig. 13). The thickness of the oxide layer is 1 μm after 24 hours and 2.5 μm after 240 hours of annealing. Changes of the chemical composition of the bulk material under the oxide layer could not be detected.

As mentioned before, the pore cavities that may be found in the inner part of the sample bulk contain small oxide particles of yttrium, titanium and aluminium (Fig. 14, Fig. 15).

4. Summary

The effects caused by annealing at 1100 °C in all three environments are predominantly connected to surface reactions of the samples. No distinctive changes have been found in the matrix in the core of both samples with the exception of MA956 pores which is solely result of the high temperature regardless of the environment. Table 3 gives a summary of the chemical changes in at surface regions – e.g oxide containing layers or depleted layers and the microstructurally

![Fig. 8. Surface of MA956 after 240 hours annealing in vacuum.](Image)

![Fig. 9. MA956, 240 hours annealing in vacuum, general overview of the sample showing pores.](Image)
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Fig. 10. Surface of MA 6000 after annealing 240 hours in hydrogen.

Fig. 11. Detail of oxide layer on MA6000 after 240 hours annealing in hydrogen.

Fig. 12. Surface of MA 956 after annealing 240 hours in hydrogen.

Fig. 13. Detail of oxide layer on MA956 after annealing 240 hours in hydrogen.

Fig. 14. MA956, 240 hours in hydrogen, general overview of the sample showing pores.

Fig. 15. MA956, 240 hours in hydrogen, detail of pore on fracture surface.
adjacent subsurface layers. All these values were measured by EDS using signal integration along a 150 µm long line parallel to the surface and only the major constituents with highest changes are shown.

### 4.1 Summary for MA6000

During air annealing experiments MA6000 produces an oxidation layer formed by aluminum oxide and nickel matrix particles, which oxidize eventually too after longer exposition times. The spalling and peeling off of this layer is the main reason for the weight loss of air annealed samples. Under the oxidized surface a pores containing zone is created. The pores have distinguished angular shapes and contain fine particles with higher titanium, aluminum and yttria content. When annealed under vacuum, MA6000 shows decrease in sample weight due to evaporation of chromium from the material. The formation of angular pores with Ti, Al, Y containing particles has been found too. Thin layer of matrix on the surface has been recrystallized. Annealing in hydrogen formed a thin uniform aluminum oxide layer with small other oxides particles.

### 4.2 Summary for MA956

MA956 exhibits excellent oxidation resistance behaviour with very low weight change. When annealed in air and hydrogen, an aluminum oxide protective layer containing fine particles, likely yttria and titanium oxides was formed on the surface of the material.

Annealing under vacuum resulted in evaporation of iron and chromium from the matrix material. The material thus exhibited significant weight loss. Large number of fine particles with high content of aluminum, titanium and yttrium remained on the surface of the sample.

All methods of annealing without exception resulted in porosity of the sample material. This porosity is not related to any of the observed surface related phenomena. The resulting pores have an oval shape elongated in the rolling direction of the material and their inner surface is covered with yttrium, aluminum and titanium oxide particles. With longer annealing time, some of the pore cavities were found to be partially filled back by matrix material.

### 5. Discussion

The types of pores identified in the examined samples differ in several parameters. In view of the position of the pores, it is possible to observe pores open to the surface, below the surface of the metal and within the mass of the material without apparent connection with the subsurface region.

In terms of shape, the pores may be divided according to whether they retain the shape of the particle in place of which they have formed or no parent feature in the microstructure is apparent.
5.1 Pores from vacancy condensation

Annealing of MA6000 gave rise to pores with a characteristic geometric shape in the regions below the formed oxide layer. On fracture surface, small particles with high content of yttrium, titanium and aluminum can be seen in the cavities of the pores, copying sharp-edged particles that are the parent structure of the pores (Fig. 16). Gonzales-Carrasco et al. [14] published a pore formation study in MA6000 during annealing in air and as a cause determined the condensation of vacancies entering the material from the oxidizing surface.

Fig. 16. Angular pores on fracture surface of MA6000 annealed for 240 hours at 1000°C in air.

Pores are formed by evaporation of matrix particles having a higher content of preferably oxidizing elements - titanium and aluminum. The evaporation of these particles begins at their boundary, which is probably already decorated by yttrium oxide particles from the powder manufacturing process. This is consistent with the results [3, 8, 9] where sites with a higher concentration of fine particles of yttrium oxide have been identified as preferred for the formation of cavities during annealing. The increasing depth of the area affected by the formation of cavities in time indicates a diffusion-controlled reaction. The mechanism of cavity formation is related to the technology of powder production and mixing and compaction.

Another observed fact is the area without visible pores that separates the oxidized surface and the area affected by the formation of cavities. The mechanism of formation of this once again homogeneous layer can be seen in a combination of vacancy transport deeper into the material layers, where larger cavities are formed than close to the surface and in the recrystallization of the surface area of the metal matrix.

It has been reported, that with a sufficiently long annealing time (hundreds of hours in a row), the pores are sequentially closed with matrix material [14]. This phenomenon was not convincingly observed on the MA6000 samples in this study.

5.1.1 Pores from gas molecule condensation

MA956, created cavities unrelated to the surface reactions of the material. The cavities in all samples - i.e. annealed in hydrogen, air and vacuum - had common characteristics. The cavities were always of oval or ellipsoidal shape with the longer axis in the direction of extrusion and rolling of the material. The internal surface of the cavities was always covered with fine particles of high content of yttrium, titanium and aluminum (e.g. Fig. 15). When observing samples annealed for 240 hours, some pores were filled with material with composition identical to that of the matrix. A likely explanation for this phenomenon is that the cavity is filled during longer annealing times.

At the fracture areas, filled or partially filled pores may be observed (Fig. 17). Two possible mechanisms were discussed in connection with pores in ferritic ODS materials. The more frequently mentioned mechanism explains the formation of cavities by condensation of the gas molecules used in the milling and mixing of powders, most often argon. The second proposed mechanism aims...
at relaxation of stresses introduced into
the material during extrusion.

By combining both approaches - which
are probably not mutually exclusive - it would
be possible to arrive at the following combined
pore formation mechanism in MA956:

During extrusion and rolling
of the material, the metal matrix is deformed
while the oxide particles in the material do not
deform. In the manufactured material, the oxide
particles are stressed by pressure while
the surrounding matrix by tensile stresses.
At the same time, argon atoms originating from
the protective atmosphere are dissolved
in the metal matrix [6, 8].

By exposing the material to high
temperature, the internal stresses in the matrix
will be relaxed by metallic atoms diffusion
on the oxide-matrix interface. This creates tiny
cavities, the free surface of which
is an opportunity for argon atoms to leave
the metal lattice. Thus both mechanisms can act
simultaneously. Cavities are first formed
by stress relaxation, because experiments with
ferric ODS materials that have not been
subjected to the appropriate deformation (about
10-15%) do not show porosity, although powder
milling takes place in an argon atmosphere.
Neither of the mechanisms directly explains
the presence of large number of yttrium-rich,
titanium and aluminum particles on the pore
surface.

5.2 Creation of new layers on samples surface.

The creation of new layers on the surface
of materials is an essential feature of corrosion-
resistant materials that facilitate the passivation
in aggressive environments [16, 17]. For the two
material under examination here layers
of Cr2O3, Al2O3, or their mixtures may
be expected to form.

5.2.1 Oxide surface layers during air annealing

On the MA6000 a Cr2O3 oxide layer
is formed first while layer of metal matrix
is retained under this outer layer, and only
beneath this mixed layer an aluminum oxide
is formed. Considering the chemical composition
of MA6000, both oxides -chromium and aluminum-
might protect the material [18].
However, long-term exposure times at high
temperatures will leave a more stable alumina
on the surface of the material. An increased
exposure time at high temperatures eventually
leads to the loss of the outer layers. This results
in weight loss of the material. The ultimate result
of long-term oxidation of MA6000 is the alumina
protective layer.

MA956 shows a typical property
for ferrite ODS reinforced with yttoria oxide
dispersion - selective formation of only alumina
on the surface upon exposure to the oxidation
environment [12, 21, 22]. This also caused
weight gain during the annealing [13, 19, 20].
The probable cause of only Al2O3 production
is a complex mechanism that shortly after
the first oxide layer is formed, yttrium atoms
diffuse into the oxide layer at the metal oxide
interface which prevents metal atoms diffusion
through the oxide towards the oxidation
atmosphere. Thus only transport of oxygen ions
through the layer towards the metallic substrate
is possible and the oxide molecules are formed
at the metal oxide interface. Experiments have
shown that increasing the yttrium concentration in the material (to 1 wt. %) increases the permeability of the oxide layer for oxygen ions meaning faster oxidation. At very low concentrations of yttrium, rippled and broken layers of alumina are formed.

5.2.2 Oxide layers created in hydrogen atmosphere

During annealing in hydrogen, layers of aluminum oxide with a thickness of units of micrometres have been produced on both materials. The formation of these layers was probably caused by an insufficient purity of the hydrogen atmosphere, which contained a small amount of oxygen, or air humidity.

The mechanism that caused also MA6000 that predominantly grows chromium oxides to create a very compact aluminum oxide in this atmosphere may be related to the stability of both oxides. $\text{Al}_2\text{O}_3$ is thermodynamically more stable, therefore, even when chromium oxide has formed on the surface of the samples, it is likely to be reduced and the oxygen reacted with aluminum to a more stable alumina. This hypothesis is also supported by the measurements of chemical composition profiles on the samples. These show a decrease in the content of chromium in the substrate material under the thin layer of $\text{Al}_2\text{O}_3$. This suggests a proposed mechanism where either chromium oxide is formed, decomposes and chromium atoms evaporate out of the material while oxygen atoms form aluminum oxide.

Considering that hydrogen of 99.99% purity was used in the experiment, at 100 kPa of the atmospheric pressure, the 0.01% of impurities gives partial pressure of 10 Pa for impurities. It is very likely that this included also some humidity that caused the oxidation in atmosphere consisting in vast majority of hydrogen.

6. Conclusions

Samples of two ODS materials were subjected to annealing at 1100°C in three different environments – air, vacuum and hydrogen.

In MA6000 the voids after air exposure have been observed in a sub-surface zone, after vacuum exposure two types of voids were identified one in the base material and one reaching the surface. In MA956 after all exposures only voids in the middle of the specimen were observed. The formation of these voids has been studied and possible mechanism is proposed.

Mainly chromium oxides have been identified on MA6000 surface after air annealing, these did not adhere well to the surface of the samples, hence the mass loss. On MA956 aluminium oxides formed a uniform, well adherent layers, hence the mass gain during oxidation. In case of hydrogen exposition, both materials developed aluminium oxide layer with titanium particles embedded in it. This is put into connection with possible minor contamination of the hydrogen atmosphere.

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