HOMOGENIZATION OF A MIXTURE OF
DISPERSION-HARDENED NICKEL AND CHROMIUM
POWERS

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In investigational work on new alloys intended for prolonged service in aggressive environments under
the simultaneous action of high temperatures and heavy loads the principal aim is to attain maximum pos-
sible structural stability and oxidation resistance. In recent years materials scientists have developed a
radically new method of increasing the heat resistance of metals and alloys, namely dispersion hardening
[1]. The introduction into a metallic matrix of fine particles of a second, usually nonmetallic, phase en-
ables materials to be produced which remain structurally stable up to temperatures close to the melting
point of the matrix metal and are in this respect markedly superior to standard age-hardening alloys.

Basically, the difference between dispersion-hardened and age-hardening alloys is that the presence
of strengthening particles in the structure of dispersion-hardened materials is not linked with their vari-
able solubility in the matrix metal, as is the case with age-hardening materials. Clearly, dispersion hard-
ening would appear to show great promise for the production of materials of high heat and oxidation resis-
tance based on the 80% Ni-20% Cr alloy. Some investigations in this field have already been carried out,
but until now use has been made almost invariably of orthodox powder metallurgy techniques, such as the
mixing of starting Ni-Cr alloy and hardening phase powders, pressing of the powder mixture, and final
heat and thermomechanical treatments of the resultant compacts [3, 4]. However, the mean particle sizes
of the metallic matrix and hardening phase powders used in these investigations were extremely large,
0.5-1.0 μ, and consequently the structure obtained was very far from ideal. According to [5], in an ideal
case the particles of the hardening phase should be 50-100 Å in size and the interparticle spacing on the
order of 0.1-0.2 μ.

At present rods, tubes, and sheets are already being produced abroad (United States) on an industrial
scale in dispersion-hardened nickel (TD Nickel) and Nichrome (TD Nichrome), which are hardened with
very fine thoria particles [6, 7]. In the Soviet Union two dispersion-hardened nickel alloys, VDU-1 and
VDU-2, have been developed, whose properties are comparable to those of TD Nickel [8, 9]. In the present
work a study was made of a process for the production of a homogeneous Kh20N80 (80% Ni-20% Cr) alloy
containing hardening phase particles resembling in size and distribution in the matrix the thoria particles
in TD Nichrome. A homogeneous nickel-chromium alloy reinforced with dispersed hardening phase par-
ticles not more than 500-700 Å in size and spaced not more than 0.3-0.4 μ apart (by analogy with TD Nich-
rome) would be expected to be markedly superior, after appropriate thermomechanical treatment, in heat
resistance to the standard Kh20N80 alloy. Several methods of producing nickel-chromium alloys con-
taining particles of a hardening phase have already been described in the literature [10, 11].

In our study the choice fell on the method of alloying with chromium metal of a dispersion-hardened
nickel powder produced by the chemical coprecipitation of nickel and thorium salts. Starting mixtures of
nickel and thorium oxide powders were prepared by carbonate precipitation from aqueous nitrate solutions;
the resultant hydroxide-carbonate precipitate was filtered off, dried, and calcined at 800°C. The oxide
mixture was then heated in hydrogen at 800°C In order to reduce selectively the nickel oxide. The weight
loss recorded indicated that the latter had been fully reduced.

By employing the method of coprecipitation of salts of the matrix and hardening phase metals it
proved possible to obtain thoria particles of 150- to 200-Å mean size (as determined by X-ray diffraction

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Fig. 1. Kinetic homogenization curves for powder compacts: 1) 70; 2) 60; 3) 50%.

Fig. 2. Variation of degree of homogeneity with temperature and logarithm of time: 1) 70; 2) 60; 3) 50%.

TABLE 1

<table>
<thead>
<tr>
<th>No. of curve in Fig. 2</th>
<th>Homogenization temp., °C</th>
<th>Homogenization rate K</th>
<th>Free term of homogenization Eq. α*</th>
<th>Full homogenization time t, h</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1100</td>
<td>1.60</td>
<td>1.32</td>
<td>0.53(2)</td>
</tr>
<tr>
<td>2</td>
<td>1100</td>
<td>1.27</td>
<td>0.80</td>
<td>1.45(3)</td>
</tr>
<tr>
<td>3</td>
<td>1100</td>
<td>0.84</td>
<td>0.50</td>
<td>3.95</td>
</tr>
<tr>
<td>4</td>
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<td>0.66</td>
<td>0.10</td>
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</tr>
<tr>
<td>5</td>
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<td>0.15</td>
<td>31.0</td>
</tr>
<tr>
<td>6</td>
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<td>0.13</td>
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<td>7</td>
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<td>0.03</td>
<td>232</td>
</tr>
<tr>
<td>8</td>
<td>900</td>
<td>0.33</td>
<td>0.015</td>
<td>646</td>
</tr>
<tr>
<td>9</td>
<td>900</td>
<td>0.30</td>
<td>0.02</td>
<td>1860</td>
</tr>
<tr>
<td>10</td>
<td>1000</td>
<td>0.24</td>
<td>0.05</td>
<td>1.60</td>
</tr>
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<td>11</td>
<td>1000</td>
<td>0.40</td>
<td>0.90</td>
<td>1.75</td>
</tr>
</tbody>
</table>

The amount of the oxide particles was 2 vol.%, an amount which is usually sufficient for the attainment of maximum hardening.

The mean particle sizes of the dispersion-hardened nickel and chromium powders were 7 and 4 μ, respectively. Their specific surfaces, determined by the nitrogen adsorption method in a Stronelein apparatus (German Democratic Republic), were 0.83 and 0.59 m²/g, respectively.

The powders were subjected to turbulent mixing for 5 h, after which the mixture obtained was pressed by the single-ended technique without lubrication in a steel die into specimens of 12-mm diameter and 3- to 4-mm height with densities of 50, 60, and 70% of theoretical. Diffusional homogenization of the mixtures was performed for 16 h at temperatures of 900, 1000, and 1100°C in a tube furnace provided with a thoroughly dried hydrogen atmosphere (with a dew point of -55°C). The temperature was raised at a rate of 700-1000 deg C/min. The degree of diffusional homogenization was assessed in experiments by the mean concentration of the diffusing element, i.e., chromium, which in turn was determined by measuring, with the aid of x-ray diffraction photographs, changes in the lattice constant of the nickel. Error in these determinations did not exceed 0.3-0.6%.

In some experiments, particularly those relating to the last stages of composition equalization, the degree of homogenization was determined by obtaining the ratio of the half-width of an x-ray diffraction line for the specimen being investigated to the half-width of a line for a fully homogenized 80% Ni-20% Cr alloy specimen. The variation of the lattice parameter of nickel with chromium concentration (up to 20 wt.%) which obeys Vegard's rule, is given in [12].

In Fig. 1 are shown kinetic curves illustrating the diffusional composition equalization of the powder compacts. At 1100°C full homogenization was attained after 2 h. With increase in compact density the time needed for homogenization decreased because the contact surface between the powder particles was greater. This result is in accord with the findings of Geguzin [13], who established that the time required for diffusional homogenization is directly proportional to the distance r between the centers of adjacent particles of dissimilar powders and inversely proportional to the contact surface area S,

$$
\tau = \frac{r^2}{D_h}; \quad \tau = \left[\frac{V_g}{2S (D_A^2 + D_B^2)}\right]^2,
$$

where $D_h$ is the coefficient of heterodiffusion, $V_g$ is the volume of a particle of the diffusing element, and $D_A$ and $D_B$ are the partial diffusion coefficients of the components of the mixture.