J. NOELS, H. MASSAUX, L. DE WILDE
BLG 475 (Feb. 1973)

FABRICATION OF DISPERSION-STRENGTHENED FERRITIC STEEL TUBES

Summary. - This report describes the possibility of fabrication on laboratory scale, of thin-walled tubes in oxide dispersion-strengthened ferritic steels. These alloys are candidate cladding materials for fast breeder reactors and are prepared by powder metallurgy techniques. Final dimensions of 6/5.2 mm (SNR specifications) have been obtained by cold drawing extruded hollows with 30/26 mm diameter.

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Samenvatting. - Dit rapport beschrijft de fabrikagemogelijkheid van dunwandige buizen in ferrietstaal verstevigd door oxydedispersies. Deze legeringen worden beschouwd als mogelijke hulsmaterialen voor splijtstofelementen van snelle reaktoren ; ze worden bereid langs poedermetallurgische weg. Einddiameters van 6/5,2 mm (SNR specifikaties) werden bereikt door koud trekken van geëxtrudeerde buizen met diameter 30/26 mm.

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Résumé. - Ce rapport décrit la possibilité de fabrication à petite échelle, de tubes minces en aciers ferritiques renforcés par dispersions d'oxides. Ces alliages sont proposés comme matériaux de gainage d'éléments combustibles de réacteurs rapides ; ils sont préparés par métallurgie des poudres. Des dimensions finales de 6/5,2 mm (spécifications SNR) ont été obtenues par étirage à froid à partir d'ébauches extrudées de diamètres 30/26 mm.

FABRICATION OF DISPERSION-STRENGTHENED FERRITIC STEEL TUBES

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BLG 475

1. INTRODUCTION

A ferritic steel, strengthened by oxide dispersion, is developed in order to solve the problem of high-temperature embrittlement of fast reactor canning material [1, 2]. A Fe-Cr-Ti-Mo matrix has been strengthened by addition of TiO_2 particles as reported previously [3]. This report deals with small-scale fabrication of dispersion-strengthened ferritic steel tubes. The aim of this study is to prove the feasibility of obtaining final tube diameters of 6/5.2 mm according to the S.N.R.* fuel clad specifications with that type of material. This was done by extrusion and subsequent cold drawing.

2. EXTRUSION

Blended and ball-milled powder with nominal composition $Fe\text{-}13Cr\text{-}5Ti\text{-}2Mo\text{-}2TiO_2$ is poured into a mild steel can as shown by Fig. 1. The powders are cold compacted in the can at about 3 kg/mm^2 and, after fitting the plug, the whole is compacted at 900°C at 100 kg/mm^2 in the container of the extrusion press. A central hole is then drilled through the compact and the can material is removed. The remaining compact is treated in a salt bath at 1200°C and extruded with an extrusion ratio of 8/1 and a ram speed of 80 mm/s. The required force is about 500 t. A hollow of about 30/26 is obtained.

3. THERMAL TREATMENT

In order to homogenize the extruded material, a thermal treatment of 10 min at 1250°C followed by 6 h at 1100°C is given in an electrode salt bath furnace.
Annealing at 1250°C is given to initiate interdiffusion of the different metallic components. The diffusion is completed by subsequent annealing at 1100°C [4]. A prolonged treatment at 1250°C causes agglomeration of the TiO_2 particles which is harmful to the mechanical properties of the material.
Fig. 2 and 3 show the microstructure of the alloy after thermal treatment.

4. PRELIMINARIES TO COLD DRAWING

Before drawing, the hollows have to be pointed at one end [5]. This pointing consists in reducing the outer diameter over a distance of about 15 cm, sufficiently to permit the reduced portion to enter freely the hole in the draw die, so that the jaws can grip this end of the tube. The point is made by swaging after salt bath heating. The molten salt acts as lubricant during swaging.
A few hours in water dissolves the remaining salt whereafter sand-blasting or grinding [6] is applied to descale the surface.
Pickling in a 20% HNO_3-3% HF solution at 60°C followed by water rinsing completes the cleaning operation.

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* S.N.R. = "Schneller Natrium-gekühlter Reaktor", Sodium-cooled fast reactor 300 MWe-prototype

5. COLD DRAWING

5.1. Determination of the drawing reduction ratio

Tensile tests at room temperature show that the material is rather brittle ; moreover, the alloy is susceptible to an ageing effect in the temperature range 600 - 1000°C. This ageing consists of a χ-phase precipitation which considerably decreases the room temperature ductility.

To bring the material in its softest condition, a solution annealing at 1200°C followed by rapid cooling has been applied.

Table 1 shows tensile test results and hardness values of samples in homogenized, solution-annealed and aged condition.

TABLE 1

Thermal history after extrusion	YS kg/mm^2	UTS kg/mm^2	ε (%)	VPN kg/mm^2
(A) 10 min at 1250°C + 6h at 1100°C	72.8	76.3	1.6	338.7
(B) as (A) + 1h at 1200°C air-cooled	45.5	78.6	10	220
(C) as (B) × 2 days at 800°C	92	97	4.4	361.7

These data show that the material is rather brittle even in the softest condition (sample B). For this reason, only a low reduction ratio per drawing pass is possible.

Preliminary attempts with an 8 % reduction ratio were unsuccessful, but 5 % passes could be given without failure. This reduction ratio per pass has been retained for the cold drawing process.

5.2. Calculation of the number of passes

Following procedure has been applied [7] .

If $\bar{d}$ = outer diameter of the tube ;

$\underline{d}$ = inner diameter of the tube ;

d = mean diameter = $\frac{\bar{d} + \underline{d}}{2}$;

t = wall thickness = $\frac{\bar{d} - \underline{d}}{2}$;

a = cross-section = $\pi d t$;

then a_0 = area of initial cross-section = $\pi d_0 t_0$ with

d_0 = original mean diameter ;

t_0 = original wall thickness ;

and a_n = area of final cross-section $\pi d_n t_n$ with

d_n = final mean diameter ;

t_n = final wall thickness .

The total reduction is defined as $R = \frac{a_0 - a_n}{a_0} = 1 - \frac{a_n}{a_0} = 1 - K \; (K = \frac{a_n}{a_0})$

The reduction during the j^th^ pass $r_j = \frac{a_{j-1} - a_j}{a_{j-1}} = 1 - k_j \; (k_j = \frac{a_j}{a_{j-1}})$

When $L = \frac{d_n}{d_0}$ and $M = \frac{t_n}{t_0}$

then $K = \frac{a_n}{a_0} = \frac{\pi d_n t_n}{\pi d_0 t_0} = L.M.$

L and M can also be written as

$$L = \frac{d_1}{d_0} \cdot \frac{d_2}{d_1} \cdot \frac{d_3}{d_2} \cdots \frac{d_n}{d_{n-1}} = \frac{d_n}{d_0} = l_1 . l_2 . l_3 ..... l_n$$

$$\text{and } M = \frac{t_1}{t_0} \cdot \frac{t_2}{t_1} \cdot \frac{t_3}{t_2} \cdots \frac{t_n}{t_{n-1}} = \frac{t_n}{t_0} = m_1 . m_2 . m_3 ..... m_n$$

also $k_1 = l_1 m_1$; $k_2 = l_2 m_2$ etc.

If the reduction per pass (r) kept constant

$$r_1 = r_2 = r_3 = ... r_n$$

then

$$k_1 = k_2 = k_3 = ... k_n = k$$
$$l_1 = l_2 = l_3 = ... l_n = l$$
$$m_1 = m_2 = m_3 = ... m_n = m$$

or $K = (k)^n$

if r and R are fixed then k is known from $r = 1 - k$

thus $n = \frac{\ln K}{\ln k}$ (total number of passes!)

also $L = (l)^n$ and $M = (m)^n$

Since L and M are known, $l = \sqrt[n]{L}$ and $m = \sqrt[n]{M}$

$$l = \frac{d_1}{d_0} = \frac{d_2}{d_1} = ...\frac{d_n}{d_{n-1}}$$

$$m = \frac{t_1}{t_0} = \frac{t_2}{t_1} = ...\frac{t_n}{t_{n-1}}$$

thus $d_1 = l d_0$ $\qquad t_1 = m t_0$

$d_2 = l d_1$ $\qquad t_2 = m t_1$

$d_3 = l d_2$ $\qquad t_3 = m t_2$

etc.

finally $\bar{d}_1 = d_1 + t_1$ $\qquad \underline{d}_1 = d_1 - t_1$

$\bar{d}_2 = d_2 + t_2$ $\qquad \underline{d}_2 = d_2 - t_2$

etc.

The initial and final diameters of the tubes being (in mm)

$\bar{d}_0 = 30.3$ $\bar{d}_n = 6$

$\underline{d}_0 = 26.1$ $\underline{d}_n = 5.2$

$d_0 = 28.2$ $d_n = 5.6$

$t_0 = 2.1$ $t_n = 0.4$

a_0 and a_n are 185.95 mm^2 and 7.03 mm^2 respectively.

Then $R = \frac{a_0 - a_n}{a_0} = 0.9622$ (96.22 %)

With a reduction ratio of $r = 0.05$ a, $\bar{d}$ and $\underline{d}$ are calculated on a digital calculating machine IBM 360/40. The values $\bar{d}_j$ and $\underline{d}_j$ are shown by Fig. 4.

5.3. Calculation of the required drawing force

The drawing force required is given by the formula $P = A \times K$
where P = required force (in kg)
A = area reduction (in mm^2)
K = is a factor depending on the UTS of the material as shown in Table 3 [8]

UTS (kg/mm^2)	K (kg/mm^2)
40	100
50	120
60	140
70	160
80	180
90	200
100	220
110	240
120	260

Application of this formula to the first drawing pass results in $A_1 = a_0 - a_1 = 8.94$ mm^2.
For a UTS of 80 kg/mm^2 K equals 180 kg/mm^2.
Finally $P = A \times K = 8.94 \times 180 = 1609$ kg = 1.6 t.
This required force is far below the drawbench capacity (4.5 t) which makes drawing possible from that point of view. Indeed, the required force for the second and following passes after thermal treatment of the tubes will be lower because $A_1 > A_2 > A_3$ ....., and K remains 180 kg/mm^2.

5.4. Drawing practice

5.4.1. Drawing tools

The drawing method used is «stationary plug» drawing. Because rather short tubes are fabricated, dies and plugs of hardened steel could be used without galling difficulties. Die and plug are shown schematically by Fig. 5 and 6. An 18 degrees entry angle (α) for the dies was used in the first drawing tests but caused frequent failure of the tubes. Decreasing the angle to 14 degrees improved considerably the drawing results. In all tests drawing speed was kept at 2 m/min.

5.4.2. Lubrication

The first lubrication method used was bonderizing [9-10-11] and immersion in a lanoline-beewax (1/1) bath at 70°C. With that lubricant small cracks appeared in the tube wall after a few passes which led to failure. Application of «Quaker Drawn 42» lubricant gives complete satisfaction. It can be applied at room temperature without bonderizing treatment. The drawn tubes have a shining surface and the lubricant is easily removed in a trichlorethylene bath at 60°C.

5.4.3. Intermediate thermal treatment

An intermediate soft annealing was applied after each pass. However, these thermal cycles result in a χ-phase precipitation (Fig. 7) which causes surface cracking and failure. These precipitates are dissolved regulary by a solution treatment at higher temperature.

6. RESULTS AND CONCLUSIONS

Serious difficulties were encountered when applying normal drawing parameters used for commercial stainless steel tube fabrication. This resulted in surface cracking and failure during the first passes. Adaptation of the drawing practice as mentioned above yielded in successful drawing of thin-walled tubes with a wall thickness of 0.4 mm and an outer diameter of 6 mm. Fig. 8 shows an as-extruded hollow and a drawn tube at final dimensions. It has been reported earlier [3] that the short-time tensile and stress-rupture properties of these materials compare favourably with commercial austenitic stainless steels (e.g. AISI 304 and 316). On the other hand, irradiation embrittlement is much less for oxide dispersion-strengthened ferritic than for austenitic steels [12]. These favourable statements, together with the described possibility of fabricating thin-walled tubes, open real perspectives for these ferritic steels as candidate cladding materials for fast breeder reactors.

Presently, work is being pursued on the improvement of the fabrication method in view of possible application on an industrial scale. Extrusion and drawing parameters can certainly be improved in order to decrease the number of drawing passes, whereas the use of a plug or pilger rolling mill could offer an additional solution for this problem.

ACKOWLEDGEMENTS

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Fig. 1 : Plug, can, compact and extruded hollow (Met. 21342)

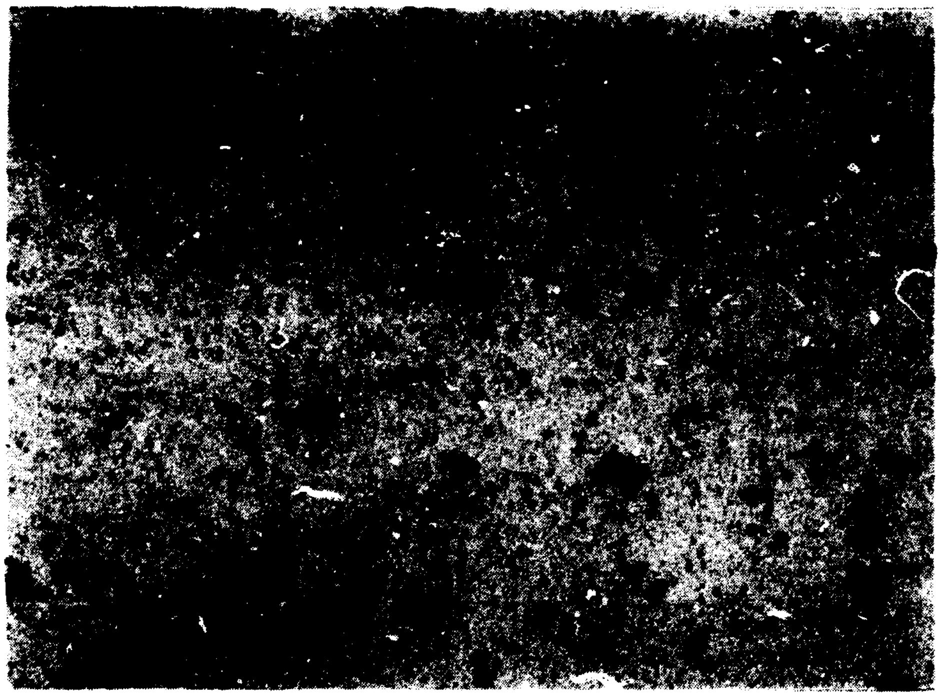

Fig. 2 : Section perpendicular to the hollow axis - unetched (x1000) (Met. 20453)

Fig. 3 : Same section but etched (x1000) (Met. 20454)

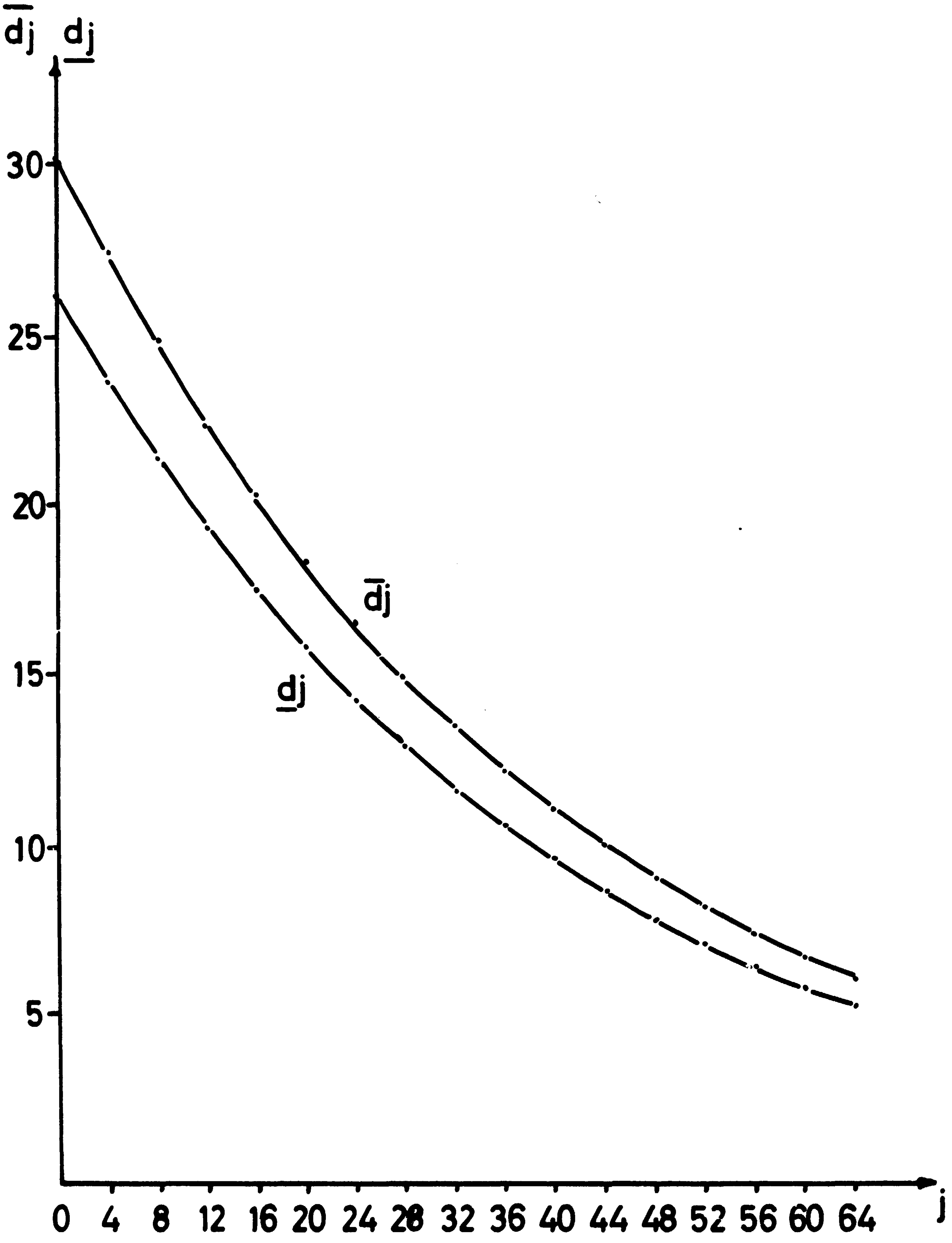

Fig.4 : Outer ($\bar{d}_j$) and inner ($\underline{d}_j$) diameters as a function of the number (j) of passes.

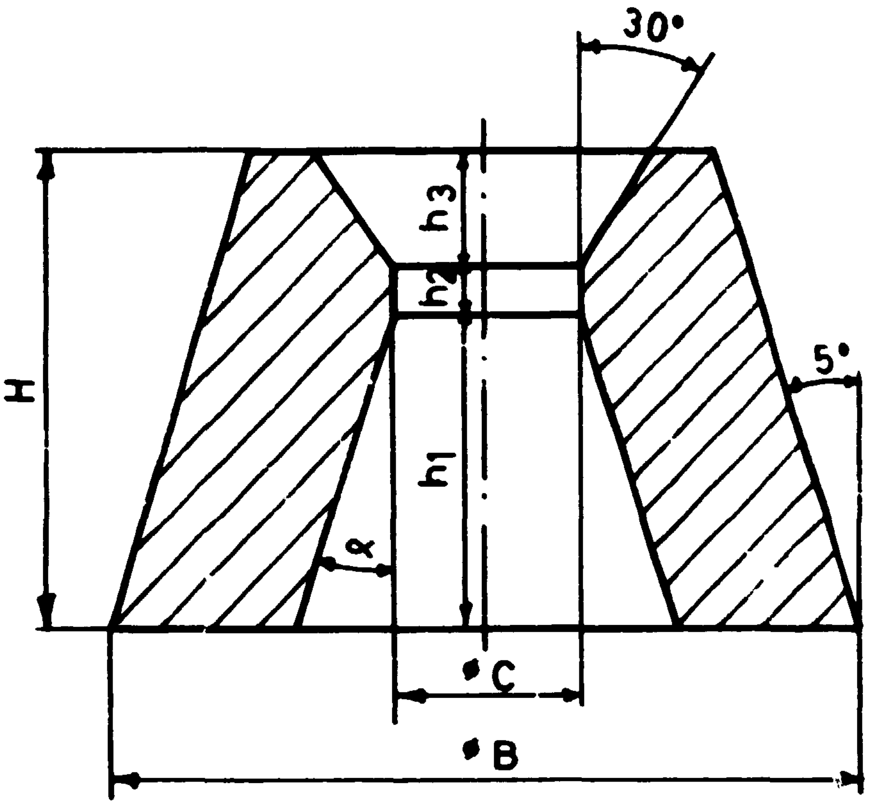

Fig. 5 : Die

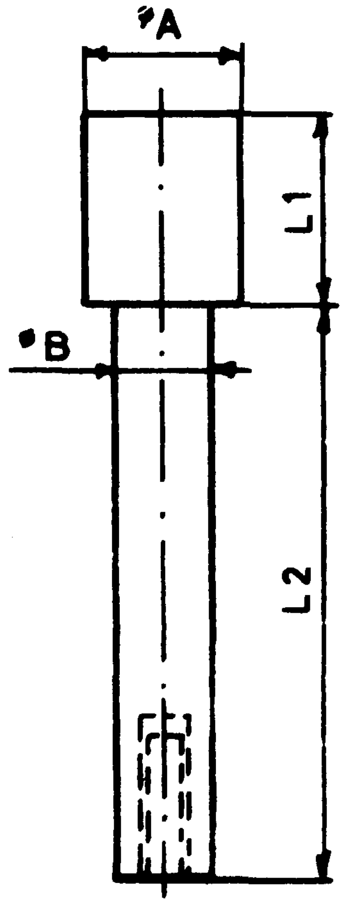

Fig. 6 : Plug

Fig. 7 : Cross-section parallel to a tube axis revealing a χ-phase precipitation in the matrix after 7 intermediate soft annealings - x1000 etched (Met. 20440)

Fig. 8 : An extruded (30.3/26.1 mm) and a drawn tube (6/5.2 mm) (Met. 20428)

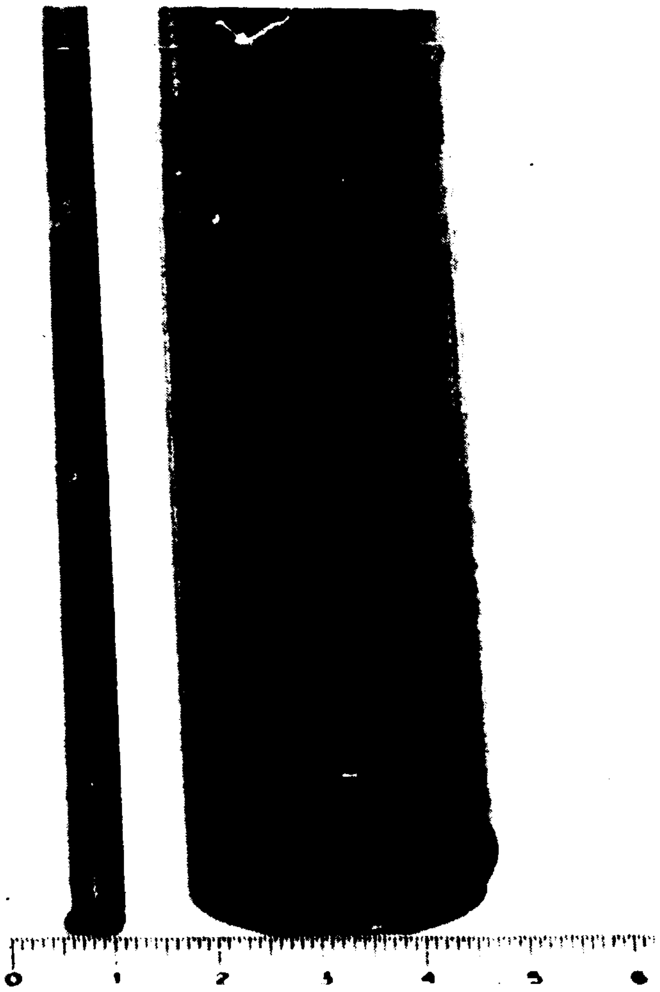

Fig. 8 : **An extruded (30.3/26.1 mm) and a drawn tube (6/5.2 mm) (Met. 20428)**

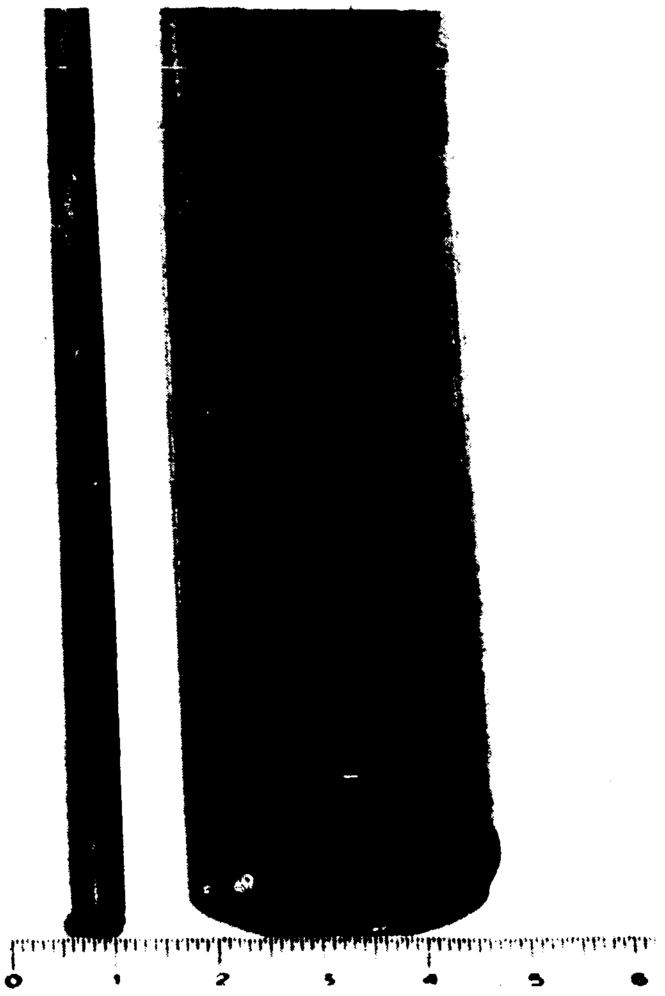

Fig. 8 : An extruded (30.3/26.1 mm) and a drawn tube (6/5.2 mm) (Met. 20428)