

HOT PLASTIC DEFORMATION OF $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ CERAMICS

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ABSTRACT

The compression mechanical properties and microstructure of the $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ ceramics in the temperature interval 825–975°C have been investigated. It has been established that three temperature intervals different in deformation phenomenology and structural changes exist. In the first temperature interval ($t=825\text{--}875^\circ\text{C}$) a value of apparent activation energy of deformation Q is equal to 500 ± 100 kJ/mol and the stress exponent n is equal to 2.5. In this temperature interval deformation occurs due to intragranular slip and climb of perfect lattice dislocations, as well as grain boundary sliding. In the third temperature interval ($t=925\text{--}975^\circ\text{C}$) $Q=1040\pm 150$ kJ/mol, $n=1.5$. The main deformation mechanism is sliding on melted grain boundaries.

The action of various mechanisms of plastic deformation leads to different structural changes. In the first temperature interval the dynamic recrystallization occurs, which causes the formation of ultrafine globular microstructure, and in the region of pre-melting temperatures a directional growth of plates through the melted grain boundaries occurs. In the intermediate temperature region a mixed microstructure is formed, which consists of both low temperature and high temperature structural components.

INTRODUCTION

At present the information on the deformation behaviour of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ (Y123) superconductive ceramics in the regions of high temperatures is very contradictory. One authors consider [1–3] that the hot deformation of this ceramics is characterized by a stress exponent n close to 1 ($n=1\pm 1.25$) and a very high value of the apparent activation energy Q ($Q=600\pm 1250$ kJ/mol) in the equation: $\dot{\epsilon} = A\sigma^n \exp(-Q/RT)$. In this case the obtained phenomenological characteristics are explained as a result of a controlling effect of the diffusion of heavy elements Ba and Y on deformation. Another authors obtained $n=2.5$ and $Q=201$ kJ/mol, but failed to give a simple explanation of the data available [4]. The common weakness of the given works is the absence of structural changes analysis during hot deformation of the Y123 ceramics.

The purpose of the present work is systematic investigation of microstructure evolution and its connection with the mechanism of plastic deformation in the Y123 ceramics within the temperature interval 800–975°C.

EXPERIMENTAL

After compaction at room temperature the cylindrical samples of Y123 ($\phi=10$ mm, $h=10$ mm) were sintered in oxygen flow at 930°C for 10 hours.

Further they were extruded to $\phi=5$ mm at 875°C . To conduct the mechanical testing on uniaxial compression the extruded rods were cut into samples of 8 mm in height. The value of stress exponent n under flow stress $n=d\lg\sigma/d\lg\dot{\sigma}$ was determined by changing the strain rate.

RESULTS AND DISCUSSION

Stress-strain curves of ceramics in the temperature interval $825-975^{\circ}\text{C}$ at $\dot{\epsilon} = 4 \times 10^{-5} \text{ s}^{-1}$ are shown in Fig.1a. Three temperature intervals differed by the phenomenology of plastic flow can be distinguished. At $T=800^{\circ}\text{C}$, due to intensive crack formation, the sample fails after $\epsilon=1.5\%$. At $T=825^{\circ}\text{C}$ resource of plasticity is about 10%. The failure is preceded by a flow stress peak which is evidently connected with cracks formation. At $T=850^{\circ}\text{C}$ the plasticity comes close to 40%. A flow stress peak is also observed at this temperature in the initial stage of deformation but it is considerably smaller than at 825°C . After the stress peak the coefficient of strain hardening θ is nearly constant up to failure and constitutes 0.2 kg/mm^2 . In the temperature interval $875-975^{\circ}\text{C}$ plasticity exceeds $\epsilon=50\%$. At temperatures $875-900^{\circ}\text{C}$ the stress-strain curves reach the stage of stable hardening immediately after the plastic flow onset. The coefficient θ does not change with increasing a strain. It is seen that in the temperature interval $850-900^{\circ}\text{C}$ at the stage of stable hardening the θ value does not almost depend on temperature.

A further increase of temperature causes qualitative changes of σ - ϵ curves. The stable stage of deformation ($\theta=0$) is achieved after small strains.

The $\ln \sigma - 1/T$ dependence is presented in Fig.1b. One can see three temperature intervals differed by the slope angle of $\ln \sigma - 1/T$. In the low temperature interval ($t=825-875^{\circ}\text{C}$) $n=2.5$, and $Q=500 \pm 100 \text{ kJ/mol}$. In the high temperature interval ($t=925-975^{\circ}\text{C}$) $n=1.5$ and the value Q rises to $1040 \pm 150 \text{ kJ/mol}$. In the temperature interval $875-925^{\circ}\text{C}$ the values n and Q are intermediate.

The microstructural investigations have shown that in the initial state the Y123 ceramics has a globular microstructure with a mean grain size of $2.5 \mu\text{m}$. As shown in [5], such a microstructure is formed due to dynamic recrystallization during hot extrusion.

As seen from Fig.2, depending on temperature, two types of microstructure are formed: globular in the temperature region $825-875^{\circ}\text{C}$ and lamellar at temperatures $925-975^{\circ}\text{C}$. In the intermediate temperature interval $875-925^{\circ}\text{C}$ a mixed type of microstructure is formed, where alongside with globular grains the islands of lamellar structure are present. With rising the deformation temperature a volume fraction of the first decreases and that of the second increases. In the intermediate temperature region a mean grain size increases due to appearance and growth of lamellar shape grains. Globular grains do not practically growth.

The energy-dispersive X-ray (EDX) analysis has shown that after deformation at $t>925^{\circ}\text{C}$ zones enriched by Ba and Cu are present in this material (mainly at triple junctions). This testifies to a partial melting of ceramics on grain boundaries during deformation. In the samples deformed below 875°C such zones are absent.

From the results obtained it follows that three temperature regions different in deformation phenomenology and microstructural changes can be distinguished. Let us consider these differences in detail.

In the temperature interval $825-875^{\circ}\text{C}$ $n = 2.5$ that is close to $n=3$ which is typical for climb of edge dislocations [6]. The apparent activation energy of deformation is far from values of activation energy of Cu diffusion ($Q_{\text{Cu}} = 240 \pm 9 \text{ kJ/mol}$ [7]) and oxygen diffusion in a tetraphase ($Q = 46 \text{ kJ/mol}$ [8]). One can suppose that in the given temperature interval a

movement of dislocations plays an important role during deformation and this movement is controlled by Ba or Y diffusion, though data about diffusion of these elements in Y123 are not available.

According to [9] after deformation of Y123 at 850°C perfect dislocations with Burgers vectors $\langle 100 \rangle$ and $\langle 010 \rangle$ are mainly observed, thus, one can suppose that deformation connected with intragranular dislocation slip is realized by sliding of $\langle 100 \rangle$ and $\langle 010 \rangle$ type dislocations on (001) planes and their climb over prismatic ones. The first is a cause of planes (001) alignment in the flow direction and, consequently, strong hardening during deformation. The climb of dislocations creates favourable conditions for formation of recrystallization nucleus and occurrence of dynamic recrystallization [10].

At the same time it seems impossible to explain the whole phenomenology of plastic deformation of the ceramics Y123 in the first temperature interval only by means of intragranular deformation. The set of experimental data indicates to the action of another mechanism of high temperature deformation, namely, grain boundary sliding. Its considerable contribution to the total deformation is attributed to the fact that the value n occupies an intermediate position between $n=2$ (which is typical for grain boundary sliding) and $n \geq 3$, when the lattice dislocations climb is a controlling factor of deformation [6,11].

A high resource of plasticity also testifies to a considerable contribution of grain boundary sliding to the total deformation. Due to limitedness of sliding systems in Y123 ceramics the Von Mises criteria is not fulfilled, thus, the compatibility of deformation is absent. In this case accommodation of intragranular slip and high plasticity are provided by grain boundary sliding.

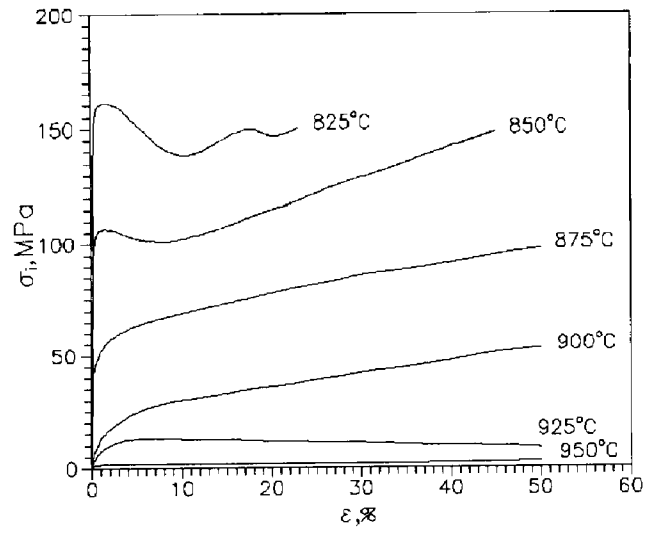
Hence, the plastic deformation of Y123 in the temperature interval 825-875°C occurs due to the superposition of two deformation mechanisms: intragranular dislocation slip and grain boundary sliding.

Another situation occurs in the third temperature region. A high value of Q , n is close to 1, and absence of hardening during plastic flow is typical for deformation of non-superconductive ceramics containing a liquid phase [12]. Thus, one can conclude that in the temperature interval 925-975°C the main deformation mechanism of the ceramic Y123 is sliding on liquid-phase intergranular layers.

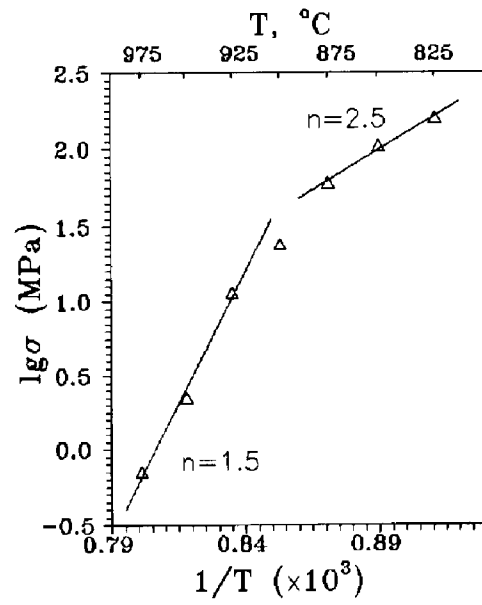
The Y123 ceramics is a precise chemical compound and a small local deviations of chemical composition can cause the appearance of liquid phases of various types [13]. The first portions of liquid occur due to the reactions: $YBa_2Cu_3O_{7-x} + BaCuO_2 + CuO \rightarrow L(e_1)$ at 898°C and $BaCuO_2 + CuO \rightarrow L(e_2)$ at 902°C. However, the liquid amount is not large, because the amount of $BaCuO_2$ is not large. A considerably larger amount of liquid is formed due to the quasiperitectic reaction: $YBa_2Cu_3O_{7-x} + CuO \rightarrow Y_2BaCuO_5 + L(p_1)$ at 933°C [13]. The possibility of melting the intergranular space in Y123 by this reaction is evidently connected with removal of copper on grain boundaries by dislocation cores. The enrichment of $\langle 100 \rangle$ type dislocations cores by Cu atoms was revealed in [14]. During deformation the separation of Y_2BaCuO_5 and $L(p_1)$ occurs, due to which the reaction becomes irreversible and after cooling traces of crystallized liquid can be observed at triple junctions.

Decomposition of Y123 and increase of diffusion paths due to space separation of products of quasiperitectic reaction in the range of grain boundaries is evidently a cause of a high value of apparent activation energy of deformation in the third temperature interval.

In the high temperature interval the dynamic recrystallization in its classic understanding is not observed. A directional liquid-phase growth of plates prevails because a lamellar grain shape is more useful for Y123.

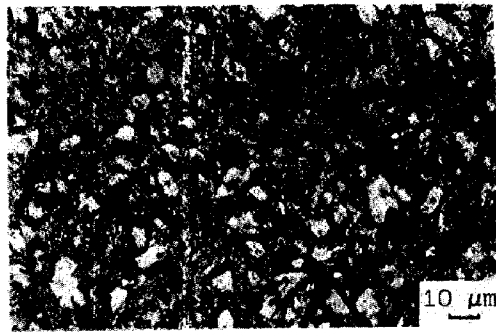


(A)

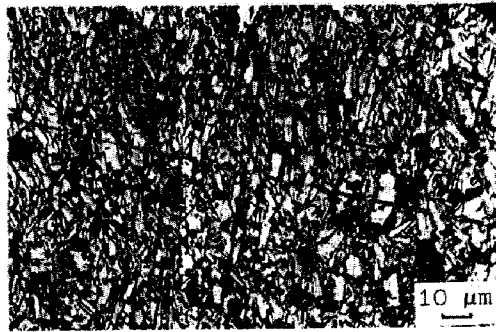


(B)

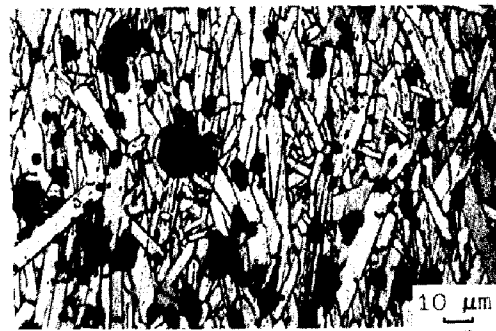
Fig.1. Mechanical properties of Y123 ceramics. a) stress-strain curves; b) $\lg \sigma - 1/T$ dependence at $\epsilon=3\%$.



(A)



(B)



(C)

Fig.2. Typical microstructures of Y123 ceramics after hot deformation with $\dot{\epsilon}=4 \times 10^{-5} \text{ s}^{-1}$ at 850°C (a), 900°C (b) and 950°C (c).

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