KFKI-1980-106

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RESEARCH
INSTITUTE FOR
PHYSICS

BUDAPEST

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To appear in the Proceedings of the Conference on Metallic Glasses: Science and Technology, Budapest, Hungary, June 30 - July 4, 1980; Paper T-25

HU ISSN 0368 5330
ISBN 963 371 752 3

АННОТАЦИЯ

Ранее проведенные исследования показали, что в сплавах металлического стекла Fe-B механизм кристаллизации зависит от концентрации бора [1,2]. В области доэвтектической концентрации кристаллизация происходит в две стадии: сперва выкристаллизовывается α-Fe в аморфной матрице, затем при повышенной температуре образуется Fe_3B. Этот процесс можно хорошо проследить с помощью магнитных измерений. В области сверхэвтектической концентрации эти две фазы не разделяются, однако, результат кристаллизации тот же самый: α-Fe и Fe_3B. Кинетика кристаллизации может быть исследована магнитными измерениями, но для более подробных исследований требуется и электронмикроскопия.

KIVONAT

Korábbi vizsgálatok már mutatták, hogy Fe-B üvegötvözetekben a kristályosodás mechanizmusa függ a bór koncentrációtól [1,2]. A hipo-eutektikus koncentráció tartományban a kristályosodás két lépcsőben következik be: először α-Fe kristályosodik ki az amorf mátrixban, majd magasabb hőmérsékleten Fe_3B képződik. Ez jól követhető mágneses mérésekkel. A hiper-eutektikus koncentráció tartományban a két lépcső nem különül el, de a kristályosodás eredménye ugyanaz, t.i. α-Fe és Fe_3B. A kristályosodás kinetikája itt is követhető mágneses mérésekkel, de részletesebb vizsgálatokhoz szükség van elektronmikroszkópra is.

ABSTRACT

It has been shown that the crystallization mechanism in Fe-B amorphous alloys depends on the boron concentration [1, 2]. The kinetics of crystallization has been followed by magnetic measurements and by electronmicroscopic investigations in both hypo- and hypereutectic concentrations.

INTRODUCTION

Evidence shows that the crystallization mechanism in Fe-B amorphous alloys depends on the boron concentration [1,2]. In the hypo-eutectic concentration range, crystallization takes place in two discrete steps: first α-Fe crystallizes in an amorphous matrix, then at higher temperatures Fe_3B is formed. This can be followed very well by magnetic measurements. At hyper-eutectic concentrations these two steps cannot be separated but the result of the crystallization process is the same, viz. α-Fe and Fe_3B. The kinetics of crystallization can also be followed by magnetic measurements in this concentration range but detailed studies require electronmicroscopic investigations.

EXPERIMENTAL

The crystallization of $Fe_{100-x}B_x$ /13<x<25/ was investigated by measuring magnetic quantities and by using a transmission electronmicroscope.

The coercive force was measured as a function of annealing time at a fixed annealing temperature and these curves were used for selecting the samples for more detailed studies. The H_c measurements were performed by an astatic magnetometer. The annealing temperature was chosen well below the crystallization temperature.

The initial permeability measurements were carried out at room temperature using the a.c. induction method on selected samples taken from the descending slope, the minimum and the ascending slope of the $H_c/t_{ann}/$ curves.

The microstructure of the samples was studied with a JEM-7 tansmission electronmicroscope. Thin films for these investigations were prepared by electrolytical polishing in a 33% solution of nitric acid in methanol at 233 K. These studies were also done on selected annealed samples or on as-quenched ones which were annealed in the chamber of the microscope. This latter method gave the possibility to observe continuously the mechanism of the amorphous - crystalline transition.

RESULTS AND DISCUSSION

In *Fig. 1* the coercive force is plotted for annealed samples as a function of annealing time.

The initial points of the measured $H_c(\tau)$ curves give the coercive force of the as-cast states. In agreement with previous results [3] it was found that H_c is higher in the ribbons prepared at higher cooling rates. The higher cooling rate the more internal stresses are quenched in. This can also be seen from the rapid initial decrease of the $H_c(\tau)$ curves for the higher cooling rate which shows the stress-relief in the ribbons. At a lower cooling rate this takes place more slowly. The increasing part of the curves seems to be connected with different mechanisms of short-range ordering. At a higher cooling rate the $H_c(\tau)$ curve rises less steeply.

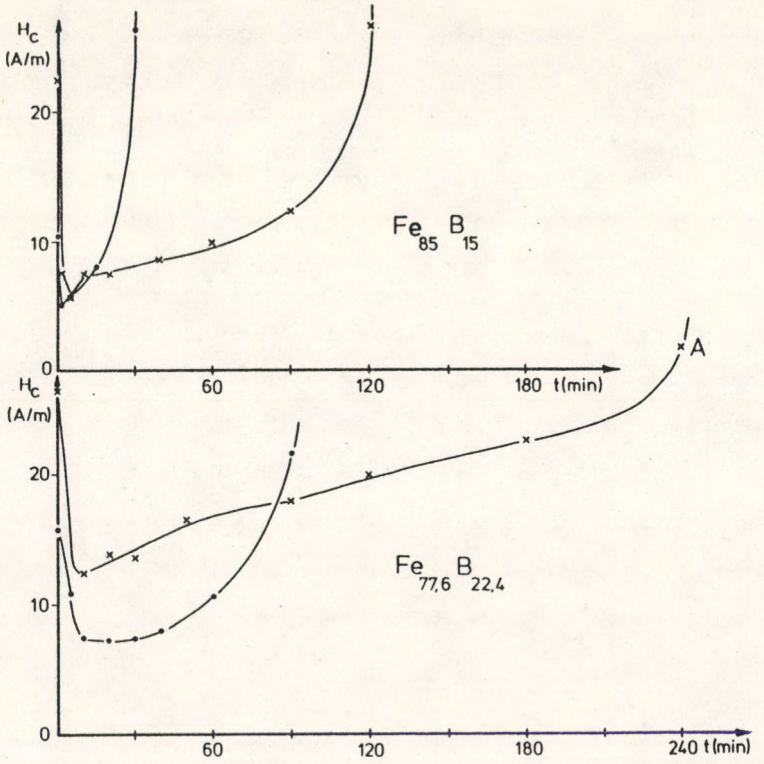


Fig.1
Coercive force measured at room temperature depending on annealing time. The samples contain 15 and 22.4 at% boron respectively. The curves for the same B content differ according to cooling rate:

-.- 6210,

-x- 12420 rev/min.

Some results of the initial permeability measurements are shown on *Fig. 2*. During heat treatment the measured permeability values show - as one might expect - a tendency opposite to the coercive force. The antiparallel changes of the two magnetic quantitites verify that the coercive field is really a measure for the mobility of domain walls in this case.

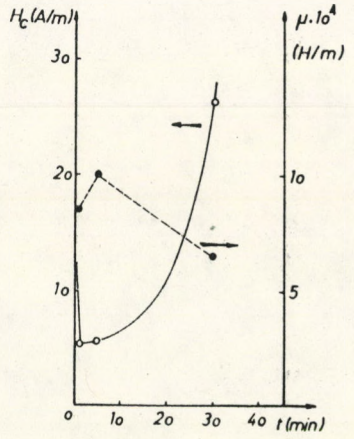
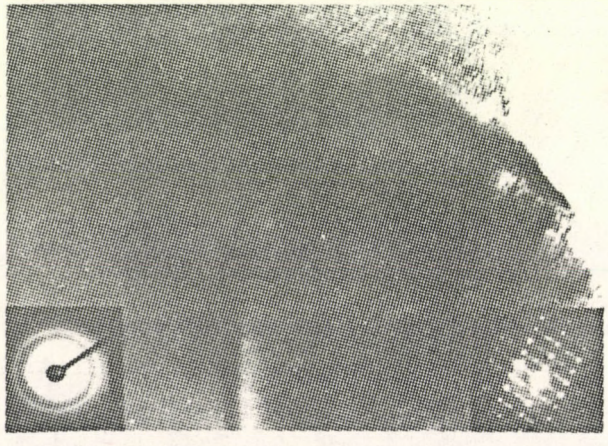


Fig. 2
Initial permeability and coercive force of $Fe_{85}B_{15}$ samples annealed at 610 K for various time durations. Cooling rate: 6210 rev/min.

Besides the magnetic measurements we also carried out electronmicroscopic investigations on some heat treated samples. It is typical that the first crystallites were detected only after a relatively long annealing time on the increasing part of $H_c(\tau)$ and have a monocrystalline character. *Fig. 3a* shows the microstructure of a heat treated sample, treated 4 hours in the astatic magnetometer /See point A in *Fig. 1*/. This monocrystal is immersed in an amorphous matrix. *Fig. 3b* shows the same part of the sample after a long period of annealing in the electronmicroscope. The electron micrographs of the crystalline region indicate that their origin is linked with the ordering process in solid solution. The previously observed monocrystal remained as it was.



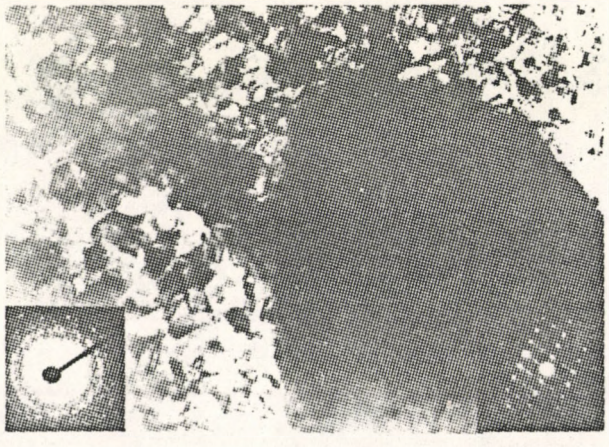
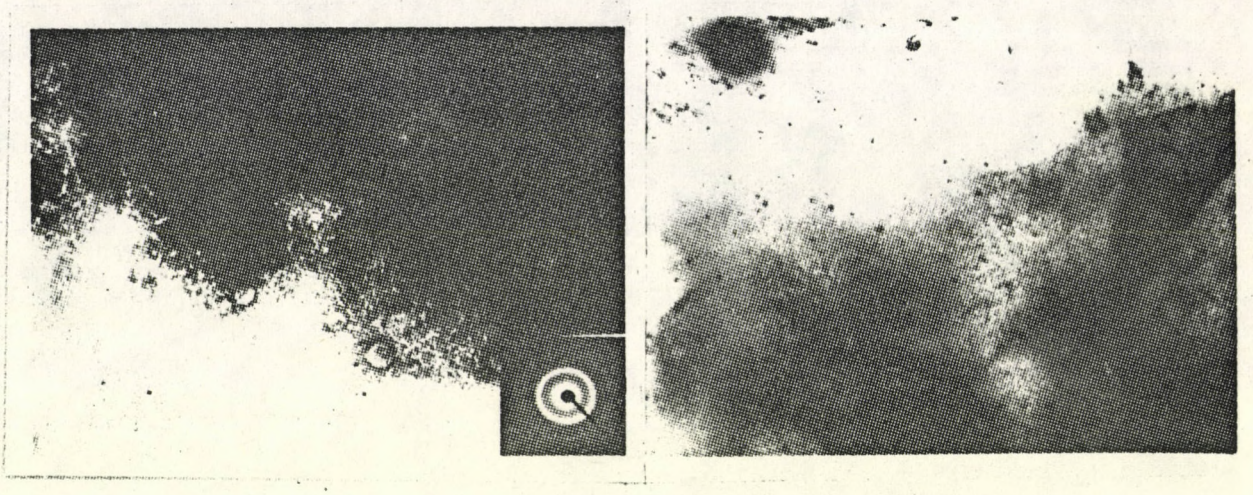
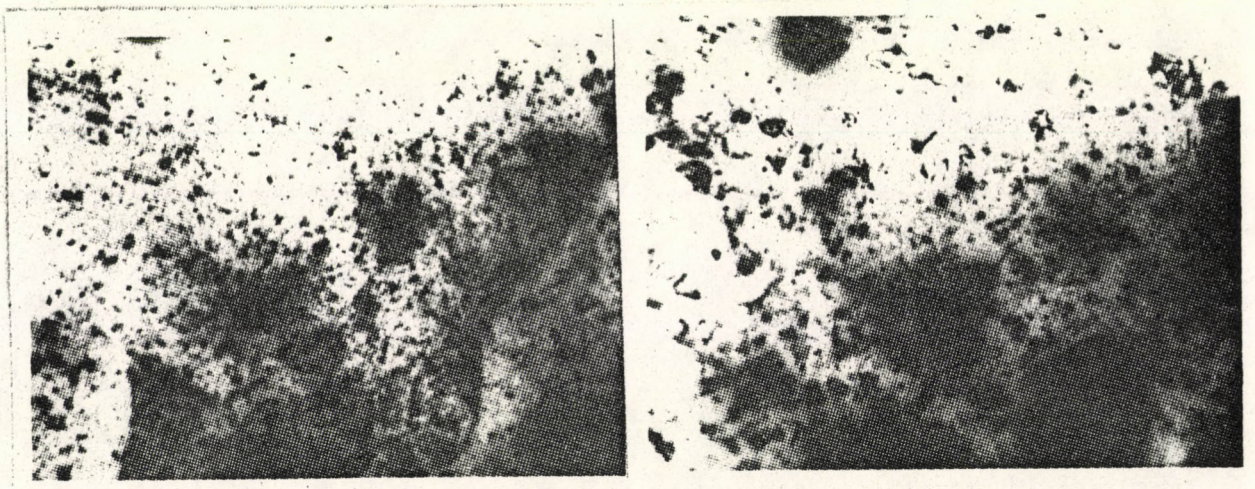


Fig.3
a/ Microstructure of $Fe_{87.5}B_{22.5}$

b/ The same sample after a long period of annealing

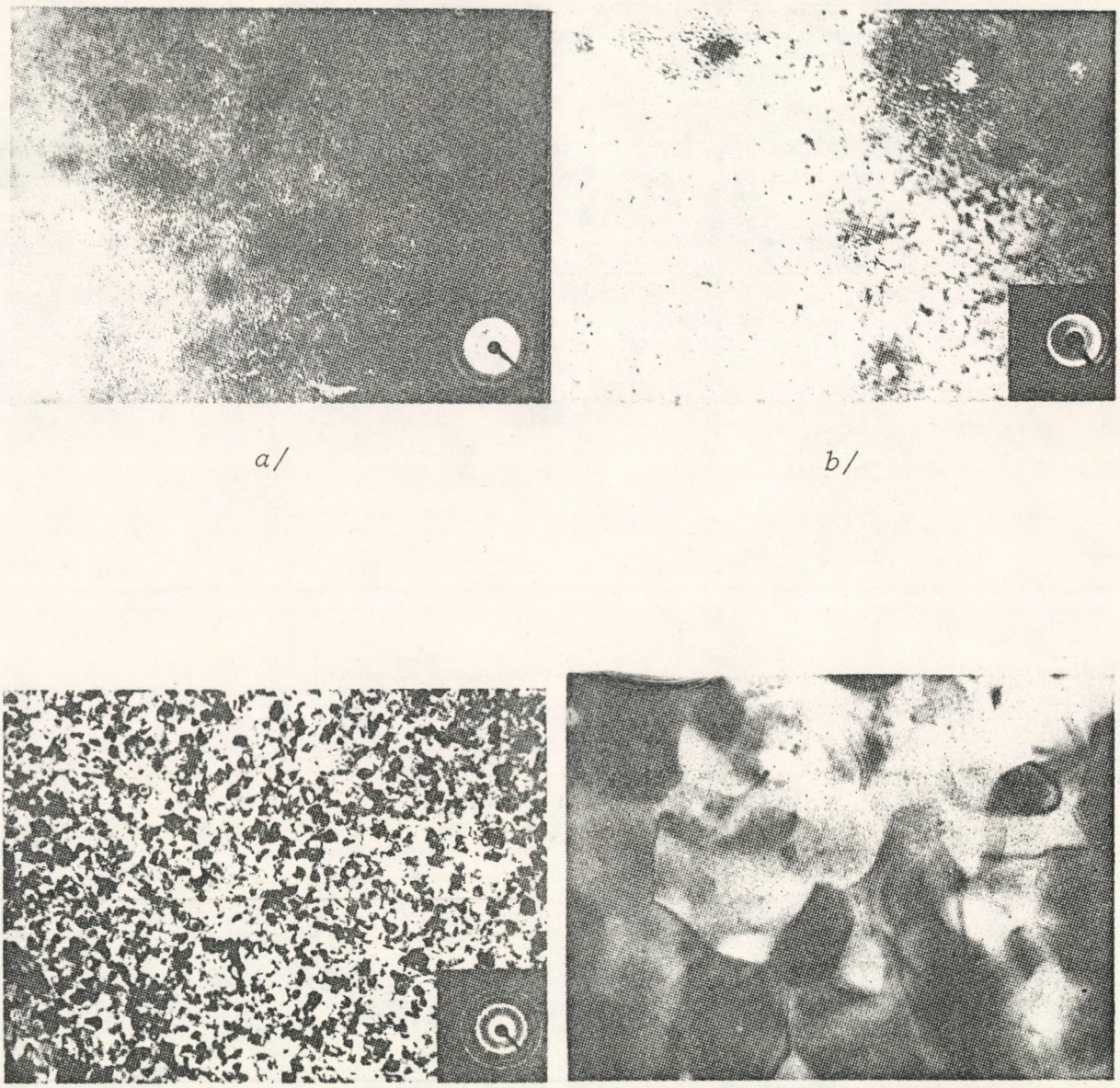


a/ b/



c/ d/

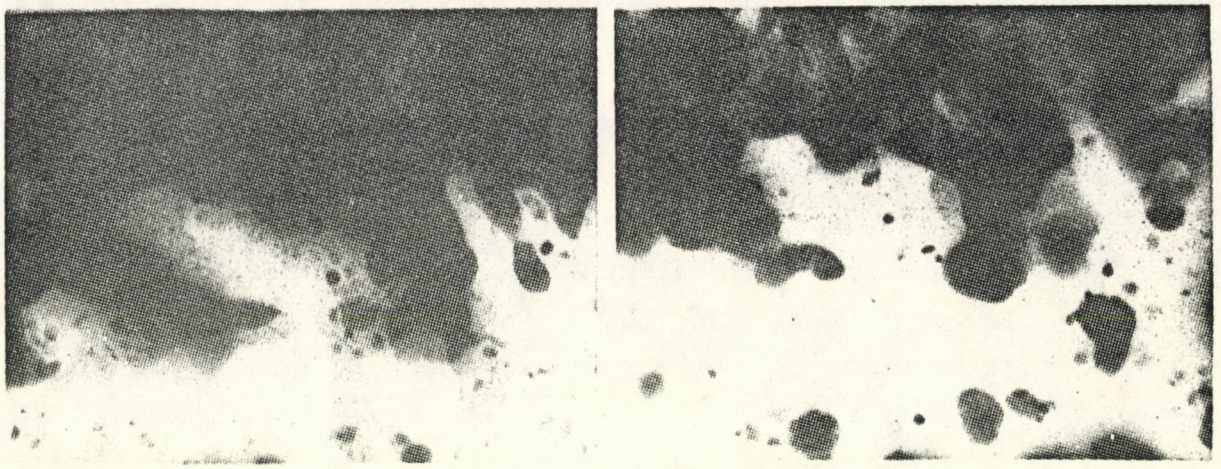
Fig. 4 Illustrations of crystallization in a sample containing 22.4 at% boron, prepared at 6210 rev/min



a/ *b/*

c/ *d/*

Fig. 5 Illustrations of crystallization showing homogeneous nucleation in 22.4 at% boron sample prepared at 12420 rev/min



a/ *b/*

Fig. 6 Illustrations demonstrating frontal movement of phase boundary during homogeneous nucleation type of crystallization

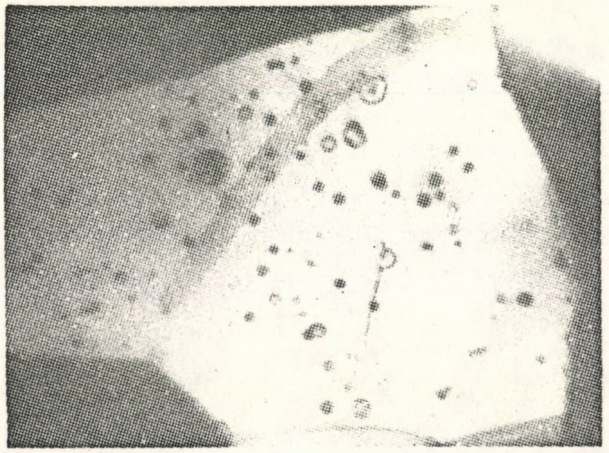


Fig. 7

Small spherical particles of Fe_3B in amorphous matrix

In the hypereutectic concentration range various mechanism were observed in which two phase decompositions of the amorphous matrix /to α-Fe and Fe_3B/ could be detected. These were investigated in-situ, in the chamber of the microscope. The crystallization may begin by heterogeneous nucleation connected with lamellar growth of nucleation centres /from "holes" already existing in the amorphous state/, after that the Fe_3B lamellae

will coagulate in the α-Fe matrix. It seems that this mechanism is probably influenced by surface diffusion */Fig. 4/*.

The second observed mechanism was the forming of polyhedral grains by homogeneous nucleation in the amorphous matrix */Fig. 5/*. This is connected with the frontal movement of the phase boundary */Fig. 6/*. It seems that these grains form a supersaturated α-Fe crystal structure in which, moreover, grains of Fe_3B compound have been observed.

Some results show that before the crystallization of α-Fe small spherical particles of Fe_3B are formed in the amorphous matrix which persist also after crystallization of the α-Fe */Fig. 7/*.

The discussed changes in the mechanism of crystallization may be connected with some chemical micro-inhomogeneities in amorphous materials.

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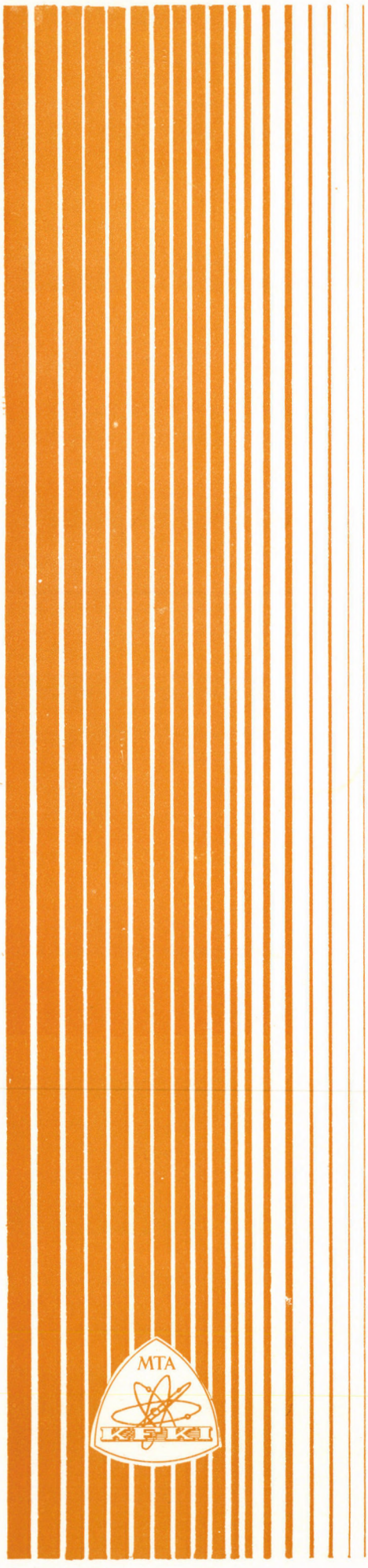

Kiadja a Központi Fizikai Kutató Intézet
Felelős kiadó: Tompa Kálmán
Szakmai lektor: Hargitai Csaba
Nyelvi lektor: Hargitai Csaba
Példányszám: 220 Törzsszám: 80-646
Készült a KFKI sokszorositó üzemében
Felelős vezető: Nagy Károly
Budapest, 1980, október hó