

## EXCHANGE INTERACTIONS IN SOFT NANOCRYSTALLINE MATERIALS

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**Abstract-** Fe rich nanocrystals embedded in an amorphous magnetic matrix have been shown to exhibit outstanding soft magnetic properties. The cause of softness is the exchange coupling between the two-phases. In this paper some effects of the exchange coupling between the nanocrystals and the matrix are reviewed.

### I. INTRODUCTION

There is no doubt about the current interest in studying the magnetic properties of multiphase systems of the part of the scientific community involved in the search for new advanced materials. The term multiphase here includes not only structural phases (ferromagnetic, antiferromagnetic, paramagnetic). This interest is based on the envisaged technological applications of such materials and also the wish to achieve a full understanding of some of the outstanding properties exhibited by these materials.

Although these materials cover a broad spectrum of characteristics and compositions, they can be classified into two major groups: non-stoichiometric systems (NSSs) and immiscible systems (ISs). Materials with various magnetic characteristics can be found within both groups. In the case of NSSs, Nd-FeB-like permanent magnets and other rare earth-transmission metal compounds generally consist of a mixture of structural phases with different magnetic properties, while Fe-based nanocrystalline materials that are characterized by a crystalline phase forming isolated nanocrystal scattered in an amorphous matrix are among the softest magnetic materials. From among the ISs, granular solids obtained from the decomposition of metastable and unstable phases (e.g., Fe-Cu and Co-Cr alloys) are an example with enormous interest as nanostructured systems with potential applications in giant magnetoresistance devices.

In this paper we firstly introduce a new simple theory which accounts for the two-phase character of the soft Fe rich nanocrystalline materials. The more remarkable point deals with the role of exchange carrier played by the amorphous matrix. Finally, we outline the more important experimental results obtained from the thermal dependence of coercivity and the measurement of the Curie temperature of the matrix in samples with different crystallised volume fraction. It will be shown that the ferromagnetic to paramagnetic transition of the amorphous matrix leads to a dramatic increment of coercivity. It is also reported that the Curie temperature of the matrix is higher than that corresponding to an amorphous sample of the same composition, thus indicating that the presence of crystallites enhance the exchange interaction within the matrix. Let us start by introducing the reasons why a two-phase model becomes necessary.

As observed by Yoshizawa et al. [1] for the first time the

Fe rich nanocrystals are soft two-phase materials which can be obtained by crystallization of conventional FeSiB amorphous alloys with small addition of Cu and Nb. The magnetic softness has been explained by Herzer [2] and is related to the ratio of the exchange correlation length (or domain wall thickness),  $L$ , to the orientation fluctuation length,  $\delta$ , of randomly distributed local easy axes, which in this case is the average crystallite size. For  $L \gg \delta$  the macroscopic structural anisotropy averages out and the domain wall can move without hindrances. Moreover, for a critical crystallized volume fraction,  $x$ , the average magnetostriction vanishes; thus magnetoelastic contributions to the macroscopic anisotropy also become negligible.

Herzer [2] has successfully applied the Alben-Becker-Chi model [3] to account for the dependence of coercivity on the nanocrystallite size,  $\delta$ . However, the random walk considerations outlined by Alben et al. were valid for a single phase system in which the exchange interactions between first neighbor magnetic moments take only one constant strength  $A$  (which is proportional to  $JS^2/a$ , where  $J$  is the exchange integral,  $S$  the spin value and  $a$  the interatomic distance). For the case of soft nanocrystalline materials there are three exchange constants corresponding to interactions between adjacent atoms of crystallites, adjacent atoms of the amorphous phase and adjacent atoms through the interface crystallite-amorphous. The simplified generalization made by Herzer has been shown to account for the main features of the dependence of coercivity on the grain size but it fails to explain the recently reported thermal dependence of coercivity [4,5] as well as the magnetic hardening observed at the very beginning stages of crystallization to a nanophase structure [6,7]. The simple phenomenological theory we are going to introduce is based on the random anisotropy approximation, but includes the two-phase character of nanocrystalline materials.

As indicated by Herzer, the relevant parameter of the theory is the exchange correlation length  $L_0$ , which for  $L_0 > \delta$  and according to ref. [3] is given by

$$L_0 = \frac{16A^2}{9k^2\delta^3} \quad (1)$$

whereas for  $\delta > L_0$  takes the value

$$L_0 = \sqrt{\frac{A}{k}} \quad (1')$$

where  $k$  is the uniaxial anisotropy constant of the nanocrystals provided they are uniaxial and  $A$  should be the strength of the exchange interactions between any pair of adjacent nanocrystals with different orientation of the easy axes. However, Herzer assumed  $A$  to be the corresponding exchange constant inside the nanocrystal. This assumption should be correct if the thickness of the intergranular amorphous matrix vanishes i. e. when the sample becomes magnetically a single-phase.

The macroscopic anisotropy  $k_0$  can be derived from random walk considerations to be

$$k_0 = \frac{k}{\sqrt{N}} \quad (2)$$

where  $N=(L_0/\delta)^3$  is the total number of crystallites lying in the volume  $L_0^3$  of uniform magnetization.  $k_0$  can be expressed, from (1), as

$$k_0 = \left(\frac{3}{4}\right)^3 \frac{\delta^6 k^4}{A^3} \quad (3)$$

The main result of this theory can be summarised as follows. For fixed  $A$  and  $k$  there is a critical length,  $\delta_0=(A/k)^{0.5}$ , which classifies the fluctuations of the easy axes orientation in two groups. a) Long wavelength fluctuations which correspond to  $\delta > \delta_0$ ; for this case, according to (1') and (2) the macroscopic anisotropy verifies.  $k_0=k$ , and b) Short wavelength fluctuations which correspond to  $\delta < \delta_0$ ; for this case the macroscopic anisotropy is the local anisotropy smoothed by the factor  $(1/N)^{0.5}$  as indicated by (2) and (3).

Notice that within Herzer's description the magnetic properties of the amorphous matrix do not exert any influence on either the macroscopic anisotropy or the exchange correlation length. There are two situations for which such approximation becomes reasonable. The former corresponds to a full nanocrystallized state ( $x=1$ ) as has been indicated previously. However, the outstanding soft magnetic properties are achieved for a value of  $x$  close to 75%, thus at this state the sample is a two-phase system. The latter corresponds to an amorphous matrix in which the exchange correlation length,  $L_{am}$ , is infinity. Notice that in this case the macroscopic anisotropy should be given by the expression (3) but multiplied by the crystallized volume fraction,  $x$ . Consequently, for low  $x$  or for temperatures at which  $L_{am}$  exhibits drastic variations a more detailed description is expected to be required.

## II. THEORY FOR TWO-PHASE SYSTEMS

### A) Phenomenological relations.

A phenomenological theory which takes into account the two-phase character of the sample can be developed by considering that the exchange coupling between magnetic moments of adjacent crystallites takes place through the amorphous matrix. Thus,  $A$  must be substituted by  $\gamma A$  where  $\gamma$  is a parameter varying between 0 and 1 and closely related to the exchange correlation length of the amorphous matrix,  $L_{am}$ . We consider that  $\gamma$  verifies the following general relationship

$$\gamma = e^{-\frac{L}{L_{am}}} \quad (4)$$

where  $L$  is the average distance between the surfaces of two adjacent crystallites. Notice that  $L$  is roughly related to the grain size and to the crystallized volume fraction through the following expression

$$L = \delta \sqrt[3]{\frac{1}{x}} - \delta \quad (5)$$

### B) Exchange correlation length of the amorphous matrix. Residual stress fluctuations.

Since we have assumed that  $L_{am}$  is finite there should be some macroscopic anisotropy in the amorphous matrix. As analysed in [8], the fluctuation length of the orientation of local easy axes in amorphous materials is about 1 nm, by taking  $k=10^4 \text{ Jm}^{-1}$  which corresponds to the magnetocrystalline anisotropy constant in ferromagnetic 3d metals, eq.(1) leads to a structural correlation length of 1 nm. Therefore, the macroscopic anisotropy is completely averaged out as can be derived from eqs (2) or (3). Nevertheless, the residual stresses give rise, via magnetoelastic coupling, to some macroscopic anisotropy,  $k_{am}$ , in the amorphous phase. The stress field is characterised by its strength and its Fourier components. As the stresses fluctuate in sign and direction the magnetoelastic anisotropy also does and the arguments used for random structural anisotropy can also be used with the local magnetoelastic anisotropy. The strength of the local magnetoelastic anisotropy due to the residual stresses can be written as

$$k_{am} = \left(\frac{3}{2}\right) \lambda_{am} \langle \sigma \rangle \quad (6)$$

where  $\lambda_{am}$  is the saturation magnetostriction of the amorphous alloy, assumed to be isotropic, and  $\langle \sigma \rangle$  is the average absolute value of the residual stresses. In FeSiB amorphous alloys, from which nanocrystalline materials are obtained,  $\lambda_{am}$  is approximately  $2 \cdot 10^{-5}$ . The average strength of the residual stresses in the as cast state has been reported to be close to 100 MPa [8]. These values lead to  $K_{am}$  of about  $10^3 \text{ Jm}^{-3}$  in the as quenched state. Therefore, the minimum wavelength,  $l$ , of the orientational fluctuation of residual

stresses, required to contribute to the macroscopic anisotropy with strength larger than  $k_0$  can be written from (3) as

$$l = \sqrt[6]{\frac{A_{am}^3 k_0}{k_{am}^4}} \quad (7)$$

where the exchange  $A_{am}$  is in the range from 1 to  $5 \cdot 10^{-12} \text{ Jm}^{-1}$ . Relation (7) indicates that for  $l$  smaller than  $30 \text{ nm}$   $k_0$  is always smaller than  $10^2 \text{ Jm}^{-1}$ . Therefore we conclude that for average strength of  $100 \text{ MPa}$  only those stress fields with wavelength larger than  $30 \text{ nm}$  contribute to the macroscopic anisotropy. Hence according to (1) the average  $L_{am}$  should be of the order of  $30 \text{ nm}$  in the as quenched state.

The composition of the amorphous matrix changes during different steps of crystallization and therefore it is a function of  $x$ . Consequently,  $L_{am}$  should show a dependence on  $x$  through the dependence of both the amorphous exchange constant  $A_{am}$  and magnetostriction  $\lambda_{am}$ . The average stress is also expected to change with the thermal treatments through either stress relaxation of the amorphous structure, for annealing temperatures below the onset of crystallization, or by the stress developed during nanocrystals formation within the amorphous matrix. In particular, after full stress relaxation, prior to the onset of crystallization the average stress of long wavelength may decrease one order of magnitude so giving rise to an increase of  $L_{am}$  above  $0.1 \text{ mm}$ .

The macroscopic anisotropy of a nanocrystalline sample consists of two components, structural,  $k^*$ , and magnetoelastic,  $k_s$ . Let us start analysing the structural component.

### C) Structural anisotropy

The contribution to the structural component is only that of nanocrystals and can be obtained from random walk considerations after taking into account the substitution of  $A$  by  $\gamma A$ , with the value of  $\gamma$  given by expression (4), and the actual  $x$ . As is easy to find, when the number of crystallites,  $N$ , in  $L^3$  is larger than 1, the exchange energy between grains varies as  $\gamma A/L^2$  and the anisotropy according to eq. (2) as  $k/(N)^{0.5}$ . Notice that  $N$  is now expressed as

$$N = x \sqrt[3]{\frac{L_0}{\delta}} \quad (8)$$

Minimization of exchange and anisotropy energies leads to the following correlation length of the two-phase system

$$L = \frac{L_0 \gamma^2}{x} \quad (9)$$

where  $L_0$  is the single phase exchange correlation length given by eq.(1), i. e. corresponding to nanocrystals at  $x=1$ .

Notice that for full crystallized samples,  $x=1$ , expression (5) leads to  $L=0$ , which value substituted in eq.(4) yields  $\gamma=1$ . Therefore, the phenomenological theory developed here contains the Herzer's model as the limit  $x=1$ . In contrast, whereas the single-phase theory restricts the dependence of magnetic properties on a parameter,  $\delta$ , the generalization for two-phase systems shows a dependence on two parameters  $x$  and  $\delta$ .

From eqs. (2), (8) and (9) the structural anisotropy can be then written for  $N>1$  or  $L>\delta/(x)^{1/3}$  as

$$k^* = \frac{x k_0}{\gamma^3} \quad (10)$$

It is important to note that the condition required for the macroscopic structural anisotropy to decrease respect to  $k$ , which for a single phase and according to (2) is  $L_0>\delta$  or, by considering (1),  $(4A/3k)>\delta^2$ , becomes for a two-phase system  $L>x^{1/3}\delta$  or according to (9)

$$\frac{4A}{3k} > \delta^2 \frac{\sqrt[3]{x^2}}{\gamma} \quad (11)$$

Therefore, calling  $\delta_0$  the maximum size of crystallites verifying  $L_0>\delta$  for a single-phase system (or  $x=1$ ), the maximum size of crystallites,  $\delta^*$ , verifying condition (11) depends on  $x$  according to (11) and (5) as

$$\delta^* = \delta_0 \frac{\gamma}{\sqrt[3]{x^2}} \quad (12)$$

Eqs. (9), (10) and (12) summarise the more important results of the magnetic behavior of a two-phase system coupled through exchange interactions and with random orientation of easy axes. As indicated by eq. (10), for a constant  $x$ , the macroscopic anisotropy varies with the sixth power of the grain size. For different  $x$  values the sixth power law becomes approximated. This dependence on  $x$  is the main characteristic of two-phase systems.

It is important to remark that in the absence of elastic stresses  $L_{am}$  would approach to infinity ( $k_{am}=0$ ), therefore  $\gamma$  should be unity and according to (10),  $k^*=xk_0$ . The existence of magnetic states with  $\gamma$  smaller than 1 implies that some stress is acting on the amorphous matrix. Consequently, magnetoelastic anisotropy exists and has to be considered in the calculation of the total anisotropy.

### III. EXPERIMENTS

Recently, Hernando and Kulik [4] and Slawska-Waniewska et al. [5] have measured the thermal dependence of coercivity for nanocrystalline samples obtained by annealing Fe-Si-B-Cu-Ta and Fe-Zr amorphous alloys, respectively. It was found that for any particular pair of  $x$  and  $\delta$  values, which depend on the annealing conditions, the coercive field shows

an abrupt maximum. The existence of this maximum evidence the presence of the factor  $\gamma^2$  in the expression (9) of L. The maximum occurs at a temperature,  $T_p$ , which is close to  $T_{am}$  for those values of x which correspond, according to (5), to  $L > 5$  nm. Nevertheless, when x increases and the corresponding distance between the surfaces of adjacent crystallites verifies  $L < 5$  nm the maximum of coercivity takes place at temperatures well above  $T_{am}$  i. e.  $T_p = T_{am} + 100^\circ$  C. This behavior can be well understood in the framework of the two-phase theory developed above.

The coercive field,  $H_c$ , roughly varies with temperature [4] as

$$H_c \propto \frac{1}{LM_S} \quad (13)$$

where  $M_S$  is the saturation magnetization. In general L increases with temperature faster than  $M_S$  decreases. Therefore, the normal thermal dependence consists of a decrease of coercivity with increasing temperature. This is, through eq.(1), the expected behavior of coercivity in a single-phase with random anisotropy. Since the exchange constant decreases with temperature (approximately as  $M_S^2$ ) slower than the anisotropy (approximately as  $M_S^n$  with  $n > 2$ ),  $L_0$  which is proportional to the ratio  $A^2/k^2$  should increase faster than  $M_S$ ; thus, according to (13),  $H_c$  should decrease with temperature. However, for a two-phase system, and as eq. (9) points out, the thermal dependence of L is given by that of the product of  $L_0$  and  $\gamma^2$ . As discussed above  $L_0$  increases with temperature. On the other hand, for constant x and d and thereby L, the thermal dependence of  $\gamma^2$  is governed by that of  $L_{am}$ , as indicated by (4).  $L_{am}$ , according to (7), should increase with temperature, reaching a sharp maximum at  $T_{am}$  and a subsequent decrease at higher temperatures. The decreasing rate of  $L_{am}$  above  $T_{am}$  is governed by the critical exponents of exchange and anisotropy at the ferro-paramagnetic transition of the amorphous matrix but it is known that  $L_{am}$  must approach to zero at  $T > T_{am}$ , independently on this rate. The decrease of  $L_{am}$  gives rise to a decrease of  $\gamma$ , according to (4) and consequently a decrease of L, as indicated by (9). As a consequence of the decrease of L,  $H_c$  increases reaching a maximum when  $\gamma$  decreases down to the value for which  $\delta$  becomes equal to  $\delta^*$ . Once this temperature is reached the grains become exchange isolated and behave as single domains only coupled through magnetostatic interactions. For increasing temperatures the coercive field decreases following the law:

$$H_c = \frac{2k}{\mu_0 M_S} \left[ 1 - 25 \frac{KT}{k\delta^3} \right] \quad (14)$$

where K is the Boltzmann constant.

At the blocking temperature,  $T_B = k\delta^3/25K$ , the sample becomes superparamagnetic. Fig.1 shows the dependence of

coercivity on temperature for the samples of  $FeCuTaSiB$  with different x. By considering that  $k=10^4$  Jm<sup>-3</sup> in  $\alpha$ -FeSi, the expected  $T_B$  ranges from  $-200^\circ$  to  $300^\circ$  C, for the experimental particle sizes, i.e.  $\delta$  ranging from 13 to 17 nm, respectively. The experimental  $T_B$  shown in Fig.1 are much larger than those expected from the values of  $\delta$ , obtained from the x-ray. This discrepancy should be attributed to magnetostatic coupling between grains [5].

The amplitude of the peak depends on x and reaches a maximum for  $x=0.15$ . Such behavior can be well understood by considering that the amplitude of the peak corresponds to the  $H_c$  value given by (14) at the temperature for which the crystallites becomes isolated i. e. at  $T_p$  for which  $\delta^*(T_p)=\delta$ .

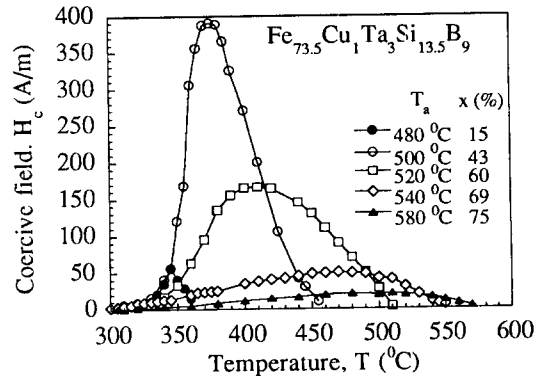


FIG. 1 Temperature dependence of the coercive field after one hour annealing at different temperatures and therefore at different crystallized volume fraction, x.

#### IV. INFLUENCE OF THE NANOCRYSTALLS ON THE MAGNETIC PROPERTIES OF THE MATRIX

Amorphous ribbons, with the following compositions, were obtained by melt spinning technique: (A)  $Fe_{96-x}B_xNb_3Cu_1$ , with  $x=19, 20, 22.5,$  and  $25$ ; (B)  $Fe_{77}B_{22-x}Nb_xCu_1$ , with  $x=3, 4, 5, 6,$  and  $7$ ; (C)  $Fe_{74.2}B_{20.2}Nb_{4.5}Cu_{1.1}, Fe_{70.7}B_{22.9}Nb_{5.1}Cu_{1.3}$  and  $Fe_{67.1}B_{25.7}Nb_{5.7}Cu_{1.4}$ .

In order to achieve different steps of the nanocrystallization process, samples of  $Fe_{77}B_{18}Nb_4Cu_1$  were submitted to subsequent heat treatments performed under an Ar atmosphere, at temperatures ranging from  $455$  to  $700^\circ$ C.

The heat treatments give rise to an ultrafine structure of  $\alpha$ -Fe crystallites, with approximately uniform grain size (10-12 nm) but increasing the volume fraction with the annealing temperature from 8 to 40%. The volume fraction of crystallized Fe, x, is accurately measured from the relative resonant area of the Mössbauer subspectra. The nominal composition of the intergranular amorphous region was determined by subtracting the relative number of crystallized

Fe atoms, obtained from  $x$ , to the composition of the starting amorphous ribbon[9].

The thermal dependence of  $M_S$  for nanocrystalline samples exhibits the typical behavior of two-phases systems. The lower Curie temperature, which corresponds to the intergranular amorphous region  $T_C^a$  ( $<T_C^{cr}$ ), can be obtained by determining the intersection point of the steepest tangent to  $M_S(T)$  curve, with the  $T$  axis and with the magnetization curve of the crystallites extrapolated down to temperatures  $T < T_C^a$ , respectively [4]. It has been observed that  $T_C^a$  increases when the volume crystallized of Fe increases. Taking into account that the Nb and B content of the amorphous matrix grows with the heat treatments as well as considering the drastic influence of Nb atoms in decreasing the Curie temperature of the amorphous alloys, we are not able to explain this result. The numerical values of  $T_C^a$  can be found in Table I.

A new experiment was performed in order to resolve this important question: Is the origin of the enhancement of the Curie temperature of the interphase any compositional changes of the amorphous matrix, or is the magnetic nature of the interphase changed by the presence of a-Fe particles?

Amorphous ribbons with the same nominal composition that the corresponding to the intergranular amorphous region were prepared (Series C) and their Curie temperature,  $T_C^{*a}$ , were measured using the same procedure. It has been found differences between the thermal dependence of the saturation magnetization for nanocrystalline samples and that corresponding to an amorphous ribbon of the same composition that the interphase. It is remarkable that the Curie temperature of the intergranular region,  $T_C^a$ , is higher than the corresponding to an amorphous ribbon,  $T_C^{*a}$ , of the same composition. The difference is large enough to discard errors in both composition estimation and measuring procedure.

Table I summarises the relevant experimental results corresponding to different nanocrystalline states of the  $Fe_{77}B_{18}Nb_4Cu_1$  alloy, obtained after different annealing temperatures. Volume fraction of crystallized Fe ( $x$ ), average grain size ( $\delta$ ), average matrix thickness ( $\Lambda$ ), and the estimated composition of the matrix are shown.

The corresponding  $T_C^a - T_C^{*a}$  for the states described in Table I are shown in Fig. 2.

TABLE I. Estimated amorphous interphase composition and relevant parameters of the  $Fe_{77}B_{18}Nb_4Cu_1$  alloy, in the as-cast state and in nanocrystalline samples enneaed at different temperatures.

$T_{ann}$ (°C)	% at. Fe E. Moss	Estimated amorphous matrix composition	$\delta$ (nm)	$\Lambda$ (nm)
as cast	0	$Fe_{77}B_{18}Nb_4Cu_1$		
455	8	$Fe_{75.5}B_{19.2}Nb_{4.3}Cu_1$	9	12
475	14	$Fe_{74.2}B_{20.2}Nb_{4.5}Cu_{1.1}$	10	11
510	22	$Fe_{73.5}B_{20.7}Nb_{4.5}Cu_{1.2}$	10	7
530	28	$Fe_{70.7}B_{22.9}Nb_{5.1}Cu_{1.3}$	10	5
555	32	$Fe_{69.5}B_{23.9}Nb_{5.3}Cu_{1.3}$	10	4
580	32	$Fe_{69.5}B_{23.9}Nb_{5.3}Cu_{1.3}$	11	4
590	39	$Fe_{67.1}B_{25.7}Nb_{5.7}Cu_{1.4}$	12	4

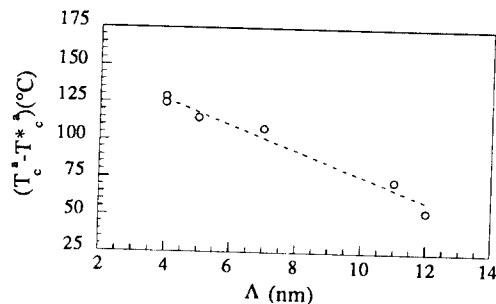


FIG. 2 Difference between the Curie temperatures of the intergranular region in nanocrystalline samples and the Curie temperatures of the amorphous sample with the same composition as the interphase,  $(T_C^a - T_C^{*a})$ , versus the interphase thickness.

## V. CONCLUSIONS

The exchange coupling between the nanocrystals is carried through the amorphous matrix. The maximum distance between crystallites required to be exchange coupled is roughly given by the exchange correlation length of the amorphous matrix. The experiments related to the thermal dependence of the coercivity have shown that strong coupling between crystallites occurs at temperatures above the Curie temperature of the amorphous intergranular region. On the other hand it has also been shown that the Curie temperature of the amorphous matrix is higher than the Curie temperature corresponding to an amorphous sample of the same composition. This Curie temperature enhancement rises with increasing crystallised fraction and is probably due to the exchange field penetration of Fe crystallites into the matrix [9].

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