# THE PECULIARITIES OF THERMOELECTRIC PROPERTIES OF BISMUTH TELLURIDE WITH IRON

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It is established that introduction of iron atoms between $Bi_2Te_3$ cleavage planes with Van der Waals forces leading to chemical bond intensification between quintets and strengthening of $Bi_2Te_3$<Fe> takes place. Moreover, the interlayer nano-structures form growing perpendicularly to (0001) $Bi_{2Te3}$<Fe> surface. Probably the interlayer ferromagnetic iron nano-structures are responsible for transition into ferromagnetic state and anomalous Hall effect (and thermo-e.m.f.) in $Bi_{2-x}Fe_xTe_3$ alloys [1].

## Introduction

The crystals on base of $Bi_2Te_3$, $Sb_2Te_3$ and $Bi_2Se_3$ present themselves the special interest both not only high-effective alloys for thermoelectricity and semi-magnetic semiconductors. The transition in ferromagnetic state is observed in such alloys as $Bi_{2-x}Fe_xTe_3$ ($0\leq x\leq 0.8$) with iron; the increase of thermo-e.m.f. *($\alpha$),* anomalous Hall effect and resistance jump at $T=T_c$ are observed [1].

Temperature $T_c$ of the transition in ferromagnetic state increases with iron content increase achieving 12K at x=0.08. The light magnetization axis is parallel to $C_3$ crystallographic axis. However, in these investigations the attention on *Fe* impurity fractal aggregate formation in Van der Waals gap and their influence on bismuth telluride peculiarities have not been paid.

The fractal aggregates (FA) form as a result of conglutination of solid particles taking place in different conditions and mediums. The models using the defined algorithms for description of motion and conglutination of solid particles during FA growth allow us to construct them and analyze both its properties and growth character. Earlier the dependence of fractal dimensionality on particle conglutination probability at mutual contact had been revealed [2-3]. Along with considered FA being in three-dimensional space FA forming on the surface (and even on crystal intracrystalline layers) present the especial interest [2,4-8]).

The physical process consideration when FA growth perpendicularly to plane at conglutination of solid particles to the substrate and growing one on it is important for us. Nowadays the nano-particles of iron and other metals on free surface [9-11] have been obtained: however the morphology hasn't been studied.

For later revealing of Van der Waals "gap" role and forming FA in them it is necessary to define the two conceptions: nano-particle and nano-reactor. First one characterizes the dimensional parameter, the second one defines the nano-particle function. Thus, for example *Fe* cluster almost loses its specific properties and approaches to metallic iron at atom number in cluster $n=15$. At $n>15$ it is cluster in dimensional meaning but it loses the "nano-reactor" properties in which properties become function of dimension [9]. In connection with experimental fact of $Bi_{2-x}Fe_xTe_3$ system transition into ferromagnetic state and possible ferromagnetism introduction of iron in this transition by nano-particles being in interlayer space let's consider the known data on iron introduction in different nano-objects [9].

The substance magnetic properties also strongly change with decrease of particle dimensions from 5-10nm [9]. For nano-clusters the non-monotonic dependence of their properties on cluster size, i.e. on atom number in it is observed [10].

In [9] the technology describing the iron cluster introduction in nano-tube is described. The growing tube forms the cluster by such way that it has the elongated form. The cross-section of such cluster corresponds in detail to tube internal diameter [11]. The nano-tubes obtained by such way have the ferromagnetism. The architecture of this nano-tube ensemble is similar to macroscopic crystal architecture in which lattice knots are occupied by iron clusters included in nano-tubes with given space periodicity [9]. It is quite possible that the given technology can be analogous one to filling technique of iron nano-particles in $Te^{(1)}$-$Te^{(1)}Bi_2Te_3$ interlayer space and can influence on $Bi_2Te_3$<Fe> ferromagnetic property change. Moreover note that magnetic property formation at cluster formation can take place either with correspondence with geometric model of solid packing or with electron shell structure.

For iron clusters the magnetic moment increase in the comparison with its value of the same atoms in massive sample is observed [10]. So for Fe atoms the magnetic moment increases up to $3.2\mu_B$ value, moreover the magnetic moment beating in the dependence on metal atom number in cluster (that is shown on fig. 4(a) of [10]) is observed. $\mu=2.2\mu_B$ value character for *Fe* atoms in massive sample takes place at the increase in cluster of atom number ($n$) up to 500 [10]. The above mentioned is necessary to take under consideration at result discussion of low-temperature ferromagnetism in $Bi_2Te_3$<Fe> semi-magnetic semiconductor revealed in [1].

Note that easily diffused impurities of transition metals can be in interlayer space of layered crystal. And in this state they should influence on mechanical, thermoelectric and magnetic properties.

From above mentioned point of view we can consider that revealing of introduction facts of Fe atoms in interlayer space, formation of nano-structures by them and their influence on $Bi_2Te_3$ is the quite actual task.

In this connection the study of iron influence having the small atomic radius on $Te^{(1)}$-$Te^{(1)}$ interlayer space morphology, on $Bi_2Te_3$ properties and its solid solution ($Bi_2Te_3$ 96 mol%- *$Bi_2Se_3$ 4* mol%) and $B_{2-x}Fe_xTe_3$ presents the interest.

## 2. Experiment technique

$Bi_2Te_3$<Fe> compound is obtained by the method of vertical directed crystallization at $950^0$K, temperature gradient $\Delta T=100^0$ and crystallization rate 1cm/h. From structural point of view $Bi_2Te_3$ – Me can be considered as intercalation one since the layers of matrix-master and "guest" (Fe) can be emphasized in them.

Here the interlayer space increase at metal atom penetration in interlayer empties is character because of weak chemical bond between $Te_e^{(l)}$-$Te_e^{(l)}$.

The electron-microscopic images are studied on atomic-force microscope (AFM) of NC-AFM mark and on electronic microscope of JSM 5410 LV mark. The roentgenodiffractional investigations are carried out on installation of Philips Panalytical (X-ray diffractometer) mark. The preparation of $Bi_2Te_3$<Fe> atomically clean surface is carried out by crystal splitting along (0001) basal plane in air before experiments.

**The obtained results and their discussion**

**3. (0001) $Bi_2Te_3$ <Fe> surface morphology**

3.1. Structure. The one of main questions relating to nature of such interlayer nano-objects is connected with their structure. Note the following experimental facts. Firstly, mainly the interlayer nano-structures (INS) have the pyramidal form without pointed end (rather similar with "hay stack"). Secondly INS having the different dimensions on height growth from (0001) surface and are parallel to each other. Thirdly, during evolution process INS remain their forms (and heights) approaching to each other; moreover the new more cubic structures having the complex form and structure. Here note that not linear dimensions but structural element dimension plays the role of characteristic one. Such structural objects are called nano-structural ones [5].

For demonstration of above mentioned the AFM images of (0001) surface of $Bi_2Te_3$<Fe> are presented. The structural elements of different heights (they are emphasized by small circles) and INS of conglutinated parts (they are emphasized by big ones) are seen on these images. It is seen that structural elements during evolution process have remained their dimensions and forms. AFM images of many (0001) surfaces of $Bi_2Te_3$-Me prove the fact of structural element conservation at the change of temperature regimes of INS obtaining.

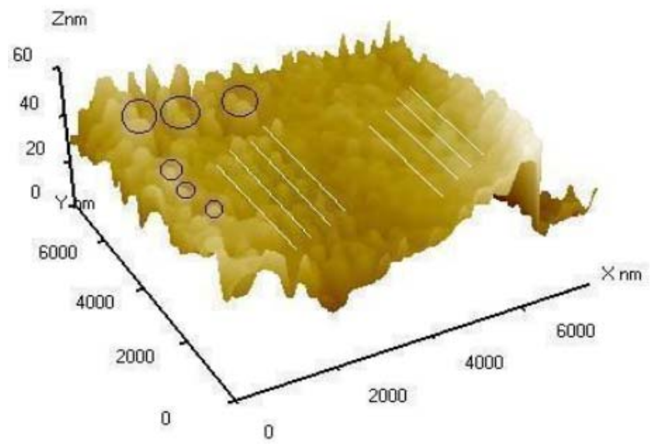


*Fig.1.* Electron-microscopic images of (0001) surface of $Bi_2Te_3$<Fe> in (3D) three-dimensional scale.

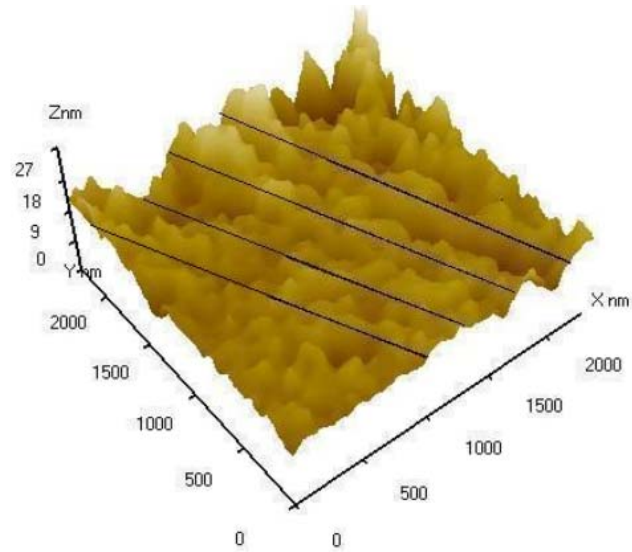


*Fig.2.* AFM-image of (0001) surface of $Bi_2Te_3$<Fe> in reduced scale (2000×2000nm).

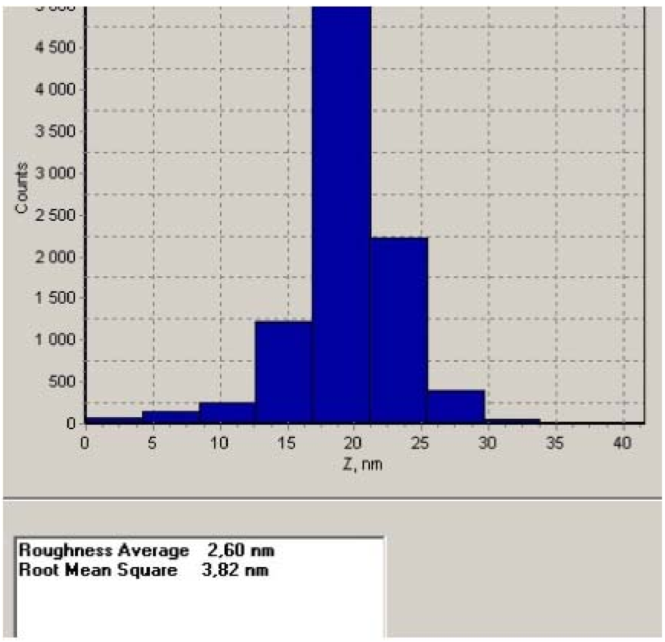


*Fig.3.* Distribution function of interlayer nano-structures on (0001) surface (on z axis) of bismuth telluride with iron (histogram).

The carried out investigations of (0001) surface in different scales are presented on fig.1 and 2 (parallel lines divide the formed "edges" of conglutinated nano-peaks). The functions of nano-particle presentations on dimensions are defined with the help of analyses of AFM images in 3D-scale, INS forms and the boundary fractal dimensions which are characteristics of object structure unevenness are analyzed. The nano-particle distribution on (0001) surface (histogram) of the same sample is given on fig.3. From image analysis it is seen that (0001) surface of $Bi_2Te_3$<Fe> consists of series of INS types. Moreover the nano-fractal dimensions fluctuate in 10nm- 30nm interval. By our opinion Fe has more less INS dimensions. From histogram it is seen that dimensions of peaks-"towers" fluctuate in 3-7nm interval; the most number of particles has the height of $h=10nm$ order. The distribution functions of unevenness on $Bi_2Te_3$<Fe> sample surface on z axis are not quiet homogeneous ones.

The histogram of distributions of separate nano-fractals (fig.3) on dimensions shows the presence of separate groups ("towers") in general ensemble with $d$=20±5nm dimensions (the "islands" by height ~35nm excepted). The presence of INS different groups can be connected with different time (and temperature) of their origin on $Bi_2Te_3$ defect surface and with peculiarities of crystallization processes (0001) on defect surface.

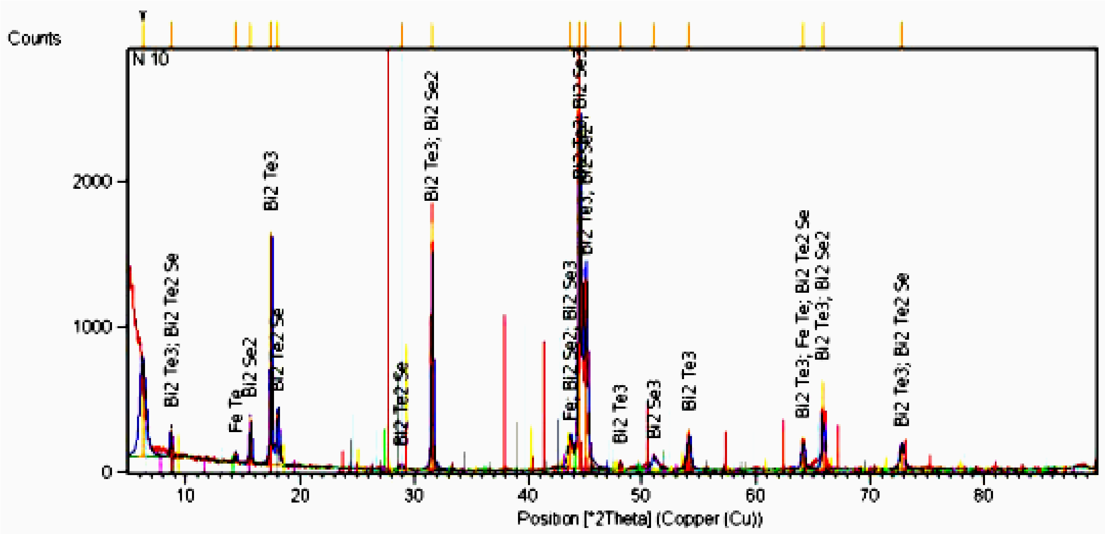


*Fig.4.* Roentgen-diffractometric image of (0001) surface of $Bi_2Te_3$-$Bi_2Se_3$)<Fe>.

As a whole, the structural elements of following types grow in $Bi_2Te_3$<metal> independently for the dependence on impurity concentrations:

- solid formations which are similar with fractal clusters of small dimensions (5-10) nm,
- single structural elements of small dimensions from 10 up to 25 nm and more high unit dimensions (>35nm)
- dense nano-structural "peaks" (see fig.1) forming the fractal surfaces.

The range of structure elements (fig.1) (which can be called elementary fractals) consisting of approached and similar to each other geometric structural figures has been distinguished by us. From the given images it is seen that nano-objects of this class are visual ones and they can be formed in 3D scale with help of structural elements (let's call it algorithm) which are similar to "hay stack" and play generator's role. One can "construct" the fractal surface with elements having different heights between $Te^{(I)}$-$Te^{(I)}Bi_2Te_3$ quintets not changing the form of such geometric fractal structural elements (FSE) and distributing them in corresponding scale.

The nano-object formation (FeTe, $Bi_2Se_3$ and $Bi_2Te_2Se$) in nano-reactor between $Te^{(I)}$-$Te^{(I)}$ at crystal growth takes place as a result of atom interaction and their aggregation at reduced temperatures (below $900^0K$). The part of iron elements "subsides" between $Te^{(I)}$-$Te^{(I)}$ $Bi_2Te_3$ layers as in nano-container not interacting with stoichiometric components that is seen from peak at $2\Theta$ =44° (fig.4). The above mentioned is proved by diffractogram given on fig.4; character peaks for $Bi_2Te_3$ are seen at angles $2\Theta$ =17; 31.5; 45°. The additional diffraction peaks from (0001splitted surface of $Bi_2Te_3$<Fe> at $2\Theta$ =15°and 64.5°c prove on nano-fragment formation from FeTe in $Te^{(I)}$-$Te^{(I)}$ nano-reactor of $Bi_2Te_3$. The appearance of diffraction peaks at angle values $2\Theta$ =16°;; 32°;; 44.5°; 52°; 73° are fixed from $Bi_2Se_2$ (fig.4).

Summarizing the investigations of AFM-images and $Bi_2Te_3$<Fe> diffractograms we can prove that series of INS: Fe, form on base (0001) surface during single crystal growth. Van der Waals "gap" of $Te^{(I)}$-$Te^{(I)}$ plays not only role of nano-reactor for FeTe, $Bi_2Se_3$, $Bi_2Te_2Se$ compounds but the role of nano-container too for Fe.

The revealing of each INS composition is practically impossible. For this purpose it is necessary to use the electronic transmission microscopy methods, roentgen emission and Mossbauer spectroscopy [1]. Nevertheless, one can take into consideration that all these structural elements are interconnected and form the stable nano-layer between $Te^{(I)}$-$Te^{(I)}$ $Bi_2Te_3$ including on all samples doped by Fe.

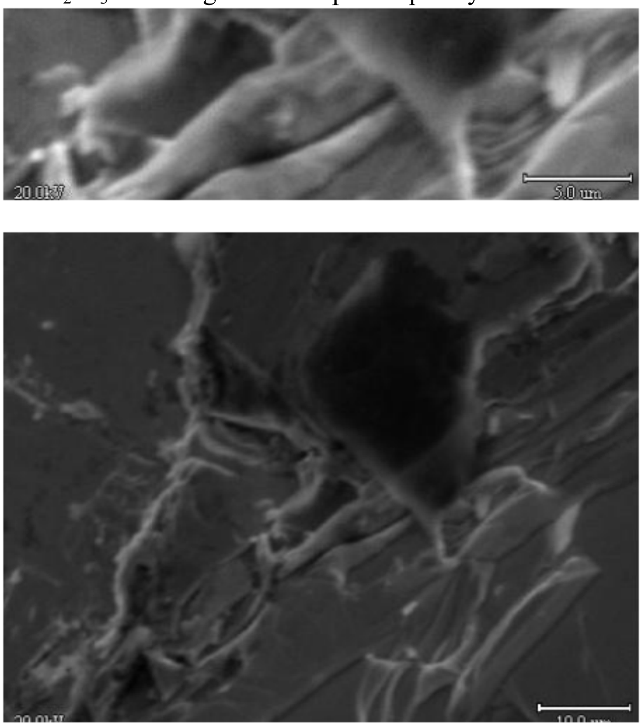


*Fig.5.* Microphoto of filamentary iron particles in $Bi_2Te_3$ matrix (between $Te^{(I)}$-$Te^{(I)}$) on (0001) plane obtained with help of JSM 5410LV microscope.

The studied interlayer nano-objects in $Bi_2Te_3$<Fe> have the complex composition and structure. INS from *Fe* having ferromagnetism is in them. The filamentary iron nano-conductors in space between $Te^{(I)}$-$Te^{(I)}$ are obtained by us. AFM-images of them are presented on photos 1 and 2 and images obtained on JSM 5410 LV electronic microscope are presented on fig.5. In right part of the given figure (fig.5) the nano-filament images in more expanded scale are emphasized separately. The Electronic-microscopic images also prove that each interlayer nano-wire seen on fig.5 can be the one-domain magnet [9] with strong magnetization anisotropy. The axis of easy magnetization is oriented along (0001) surface of $Bi_2Te_3$<Fe> (in which the nano-filaments are situated) [9]. The fact, that in all samples with Fe the transition into ferromagnetic state with easy axis along $C_3$ axis [1] (perpendicular to (0001) surface of $Bi_2Te_3$<Fe>) at $T=T_c$ is revealed, can be connected with interlayer *Fe* nano-particles (fig.1,2 and 5).

### 3.2 INS growth mechanism

The nano-layers formed on (0001) surface interact with superstoichiometric ones of *Bi, Se* and *Te* forming *FeTe* and $Bi_2Se_3$, $Bi_2Se_2$, $Bi_2Te_2Se$ also. The part of non-interacting iron atoms also subside between $Te^{(I)}$-$Te^{(I)}$ layers in free state as in nano-container.

The vacancies from under *Te* on complexes of "vacancy-impurity atoms" types can be the more possible places of accumulation and origin of nano-fragments on iron base. The places round dislocation holes and other extensive defects of block and grain boundaries, micro-gaps, concentration inhomogeneity and micro-segregation phenomena [8] can be more complex places of nano-fractal formation.

The comparison of revealed fractal formations shows both the similarity and difference in their dimensions and probably in formation mechanisms of nano-formations.

The big numbers of bound iron atoms which conserve their individuality (see fig.4) inside the given object are observed at investigation of interlayer metallic nano-particles. The "nano-fractals" term can be propagated on FeTe, $Bi_2Se_3$, $Bi_2Te_2Se$ nano-objects being inside $Te^{(I)}$-$Te^{(I)}$ layers and consisting of defined number of bound microscopic particles.

Comparing such nano-objects with known fractal clusters in [2, 4-6] one can conclude that such nano-formations present themselves the fractals with fractal dimensionality $D \sim 1{,}45$.

The formation mechanism of fractal aggregates probably is connected with filling process of places by impurities round dislocation holes, grids and vacancies under from Te on (0001) surface inside $Bi_2Te_3$. The formation beginning of fractal cells takes place during crystal growth with formation of their germs on telluride quintets: including on vacancy places being on quintets.

The total filling of the gap between $Te^{(I)}$-$Te^{(I)}$ by nano-fragments during their dimension growth takes place, as a result of which the nano-wires (see fig.5) with nano-"towers" on their surface (three-dimensional images of which we see on fig.1 and 2) form.

### 3.3 $Bi_2Te_{3-x}Se_x$<Fe> properties

The interlayer INS can be considered as the one of the factors defining the morphology of interlayer space and influencing on many properties including on thermoelectric, mechanic and magnetic ones.

Let's consider the experimental data given in the table, here $\alpha$-thermo-e.m.f., $\sigma$ is electrical conduction, *H* is thermal conduction. Also $\sigma_{com}$ is compression ultimate strength and thermoelectric quality factor *Z* ($Bi_2Te_3$96mol%-$Bi_2Se_3$4 mol%) in the dependence on iron percentage have been studied. The coincidence of maximums $\sigma_{com}$ and $Z_m$ at 0,05 weight %Fe, moreover $\sigma_{com}$ increases almost in 2,5 times; for negative branch n-$Bi_2Te_3Se_x$<Fe> ($0 \le x \le 3$) the $\sigma_{com.max}$=5.2kgf/mm$^2$ is very high strength index. The thermoelectric goodness is $Z_{max}=3.1\cdot10^{-3}K^{-1}$ at 0,05% *Fe*.

As it is seen from the table at 0,25% weight Fe the conduction sign inversion from *p* type on *n* one takes place. The hole concentration in samples ($Bi_2Te_3$-$Bi_2Se_3$)<Fe> decreases as in $Bi_{2-x}F_xTe_3$ system with growth of *x* and *Fe* begins to reveal the donor properties [1]. The thermo-e.m.f. increases from $215\cdot10^{-6}$ V/c in pure sample up to $260\cdot10^{-6}$ V/c in $Bi_{1,92}Fe_{0,08}Te_3$ with maximum *Fe* content [1].

Thus, the mechanical strength is increased in 2,5 times in comparison with undoped systems at the conservation of thermoelectric goodness on high level $Z_m=3.1\cdot10^{-3}grad^{-1}$ because of intensification of interlayer interactions in $Bi_2Te_3Se_x$<Fe> at Fe=0,05 weight %.

Introduction of iron impurity leads to electron concentration increase that is connected with total blanking of anti-structural defects of $Bi_2Te_3$. Probably, for defined concentrations the iron atoms are in solid solution in points of tellurium sublattice as neutral defects preventing to formation of anti-structural defects of $Bi_2Te_3$. The increase of impurity concentration gives the possibility of iron atom localization in bismuth vacancies and in interstices, the incompleteness of *d*-shell of which leads to electron localization on these impurity centers that is explained their acceptor influence.

Considering that defects $Fe_{Te(2)}$ are neutral ones and $Fe_{Bi}$ positions are ionized and localize the electrons on it, so the electron density displacement to the side of $Te^{(I)} - Bi, Te^{(I)} - Fe_{Bi}$ bond is possible. The corresponding displacement of electron density and small quantity of *Fe* localized in interlayer space causes to decrease of interlayer energy barrier and intensification of interlayer interaction because of overlapping of wave functions of neighbor layer electrons and impurities being between layers.

The *Fe* introduction higher than 0,06 weight% leads to gradual decrease of mechanical strength up to values less than undoped samples have. The strong behavior of electric conduction change is changed on stepless one, and thermo-e.m.f. coefficient essentially doesn't change.

Here we should note [10] that iron atom magnetic moment in mono- and bi-nuclear iron compounds is 6 $\mu_B$ meanwhile the magnetic moment of iron atom in massive ferromagnetic is equal to 2,2 $\mu_B$. The magnetic moments of *Fe* atoms being in crystal lattice points (interstices) change at their transition into interlayer space of $Bi_2Te_3$. This can be connected with possible bound change of $Te^{(I)}$ atoms of INS constructed from clusters. The formation of magnetic bonds at cluster formation can carry out in the correspondence with construction of electronic shell as in the case of alkali metal clusters [10]. The dimension decrease of INS of *Fe* in interlayer space $Te^{(I)} - Te^{(I)}$ of $Bi_2Te_3$ can lead not only to change of its magnetic moment, increase of thermo-e.m.f. but to transition of whole crystal $Bi_{2-x}Fe_xTe_3$ into ferromagnetic state moreover it is quite possible that nano-filaments can play the role of domain boundaries at magnetization.

## CONCLUSION

The formation on (0001) interlayer surface of $Bi_2Te_3$<Fe> nano-particles can be considered as evolution process the stages of which develop in following sequence:

1 iron atom introduction into places with crystal intrinsic (antistructural) point defects;

2 the further transition of *Fe* from intralayer telluride quintets into interlayers:

3 the formation between $Te^{(I)} - Te^{(I)}$ nano-particles (nano-wires) of *Fe* and its compounds with *Te* (of FeTe type).

The peculiarity of such processes is that INS forms by the way of sequential physicochemical processes accompanying by their composition change and appearance of nano-filaments.

The study of reactivity of two-dimensional structures of $Bi_2Te_3$ and *Fe* guest reveals the chemical transformation between $Te^{(I)} - Te^{(I)}$. As a result of it the nano-particles with islands – "towers" have formed on (0001) surface between telluride quintets.

The electron-microscopic and roentgenodiffractional investigations show that INS from iron and *FeTe* are realized in nano-structures with real surfaces. The "towers" formed on islands are oriented perpendicular to (0001) surfaces in high-performance thermoelectric crystal ($Bi_2Te_3$-$Bi_2Se_3$)<Fe> and have three-dimensional INS.

Van der Waals gap between $Te^{(I)} - Te^{(I)}$ quintets is two-dimensional nano-reactor in which *FeTe* $Bi_2Te_2Se$ displace.

In all cases the formed nano-structural particles can be the reason of *(Bi-Te-Se)<Fe>* crystal mechanical strength increase. The transition into ferromagnetic state of $Bi_{2-x}Fe_xTe_3$ alloys, thermo-e.m.f. increase and Hall anomalous effect observed in [1] can be connected with nano-objects consisting of ferromagnetic iron atoms.

Table

Thermoelectric properties of ($Bi_2Te_3$- $Bi_2Se_3$) solid solution doped by iron at $T$=$300^0K$

| Iron percentage, weight% | Thermo-e.m.f., $\alpha \cdot 10^{-6}, V/C$ | Electroconductivity, $\sigma \cdot 10^2, Sm/m$ | Thermal conductivity, $H \cdot 10^{-3}, Vtt/m \cdot K$ | Thermoelectric goodness, $Z \cdot 10^{-3}, K^{-1}$ | $\sigma_{com}$, $kgf/mm^2$ |
|---|---|---|---|---|---|
| 0,0 | +220 | 800 | 16,0 | 2,5 | 1,8 |
| 0,01 | +230 | 800 | 16,7 | 2,5 | 2,0 |
| 0,015 | +232 | 780 | 16,7 | 2,5 | 2,0 |
| 0,018 | +247 | 760 | 16,6 | 2,5 | 2,0 |
| 0,025 | -218 | 750 | 16,3 | 2,2 | 2,3 |
| 0,04 | -228 | 840 | 15,3 | 2,9 | 4,0 |
| 0,05 | -245 | 730 | 14,1 | 3,1 | 5,2 |
| 0,06 | -260 | 500 | 13,8 | 2,5 | 4,5 |
| 0,08 | -250 | 300 | 13,2 | 1,4 | 2,3 |
| 0,09 | -250 | 180 | 13,0 | 0,9 | 1,8 |
| 0,1 | -253 | 150 | 12,8 | 0,8 | 1,5 |


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## $Bi_2Te_3$<Fe> KRİSTALLARIN TERMOELEKTRİK XASSƏLƏRİNİN XÜSUSİYYƏTLƏRİ

Göstərilib ki, *Fe* atomları $Bi_2Te_3$<Fe> laylar arasına daxil olaraq kristalı möhkəmləndirir. Bu zaman laylar arasında yaranan nanostrukturların istiqaməti (0001) satinə perpendikulyar olur. Ola bilər ki, ferromaqnit keçidi və anomal Xoll effektində (və termoedesinin artımı) $Bi_{2-x}Fe_xTe_3$- kristallında əsas rolu aralıq ferromaqnit dəmir nanostrukturları daşıyır.

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## ОСОБЕННОСТИ ТЕРМОЭЛЕКТРИЧЕСКИХ СВОЙСТВ КРИСТАЛЛОВ ТЕЛЛУРИДА ВИСМУТА С ЖЕЛЕЗОМ

Установлено, что происходит внедрение атомов железа между плоскостями спайности $Bi_2Te_3$ с ван-дер-ваальсовыми связями, приводящее к усилению химической связи между квинтетами и упрочнению $Bi_2Te_3$<*Fe*>. При этом образуются межслоевые наноструктуры, выростающие перпендикулярно поверхности (0001) $Bi_{2Te3}$<*Fe*>. Вероятнее всего за переход в ферромагнитное состояние и аномальный эффект Холла (и рост термоэдс) в сплавах $Bi_{2-x}Fe_xTe_3$ [1] ответственны внутрислоевые ферромагнитные железные наноструктуры.

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