Evidence of Electronic Phase Separation in the Strongly Correlated Semiconductor YbB$_{12}$

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In the family of rare earth (RE) dodecaborides, YbB$_{12}$ is an archetypal strongly correlated electron system (SCES), which has been actively studied since the 1980s as a fluctuating-valence narrow-gap semiconductor.$^{[1–3]}$ Unstable valence 2.9–2.95 of Yb ions is accompanied with the metal-insulator transition (MIT), which is observed with decreasing temperature both in pure YbB$_{12}$ and in $R_{1−x}$ Yb$_x$B$_{12}$ solid solutions with partial substitution of Tm or Lu for Yb (see Ref. [4] and references therein). Moreover, at low temperatures the insulator-to-metal transition is observed in YbB$_{12}$ under pressure$^{[5]}$ and in strong magnetic field.$^{[6,7]}$ The mysterious insulating ground state in YbB$_{12}$ and similar SCES compound SmB$_6$ has attracted considerable interest from theorists, and various models have been proposed to explain the spectacular properties of this system. Some of them are based on the coherent band picture where the energy gap forms due to the strong $d$–$f$ hybridization.$^{[6,7]}$ The inversion between 4$f$ and 5$d$ bands that accompanies this process was considered as an essential characteristic of the topological Kondo insulator.$^{[10–12]}$ Other models are based on a local picture, where the electrons contributing to the Kondo screening are captured by the local magnetic moments resulting in an excitonic local Kondo bound state.$^{[13]}$ Among the exotic electronic properties recently discovered in the archetypal SCES narrow-gap semiconductors SmB$_6$ and YbB$_{12}$ with a very unusual insulating ground state and metallic Fermi surfaces, we highlight (i) quantum oscillations of magnetization and resistivity, which are the fingerprints of normal metal, and (ii) extraordinary gapless charge-neutral fermionic excitations.$^{[6,7,14–16]}$ The conclusion about the finite density of states $N(E_F)$ at the Fermi energy in the YbB$_{12}$ semiconductor was based mostly on the low-temperature ($T \leq 14$ K) heat capacity (HC) analysis, developed in Refs. $[6,7,14–16]$. On the contrary, a wide range ($T \leq 200$ K) HC study of YbB$_{12}$ allowed the authors$^{[17]}$ to separate one Einstein and two Schottky-type components at intermediate and low temperatures arguing in favor of very small values $N(E_F) \sim 0$.

The above-mentioned models are based on the postulate of stable B$_{12}$ nanoclusters as the main structural elements of the RE dodecaborides RB$_{12}$, whose structure is similar to the fcc structure of NaCl when Na is replaced by R and Cl is replaced by B$_{12}$ cuboctahedron [Fig. 1(a)]. On the contrary, it has been shown recently (see Refs. [4,18] and references therein) that the cooperative Jahn–Teller (JT) instability of the boron framework (ferrodistortive effect) is among the main factors responsible for (i) the static and dynamic fcc lattice JT distortions and (ii) formation of dynamic charge stripes in the matrix of RB$_{12}$. These fluctuating charges (stripes) lead to nanoscale phase separation,$^{[19]}$ which creates a strong inhomogeneity in the electron density (ED) distribution in the conduction band, combined with symmetry breaking in these highly entangled SCES.$^{[4,18]}$ In the Yb-based RB$_{12}$ the JT instability of the boron sub-lattice is accompanied with the valence...
instability of Yb, so that fast on-site 4f–5d charge and spin fluctuations of ED can modify the dynamic charge stripe patterns in the model SCES.\(^4\) The dynamic charge stripes (ED fluctuations with the frequency \(\nu_s \sim 240\, \text{GHz}\))\(^{19}\) were discovered previously in LuB\(_{12}\) at \(T = 50\, \text{K}\)\(^{20}\) in ZrB\(_{12}\) at \(T \sim 100\, \text{K}\)\(^{21}\) and in the Tm\(_{0.19}\)Yb\(_{0.81}\)B\(_{12}\) even at room temperature.\(^4,22\) However, the possibility of existence of these singularities in the parent narrow-gap semiconductor YbB\(_{12}\) has not been investigated up to date.

To shed more light on the nature of the ground state, the driving mechanisms of fluctuating charges and their spatial patterns in the Yb-based mixed-valence SCES, we carried out (i) x-ray diffraction (XRD) studies of high-quality YbB\(_{12}\) single-domain crystals, (ii) measurements of the low-temperature polarized terahertz response, and (iii) accurate charge transport experiments. Clear evidence of charge stripes was demonstrated by the XRD analysis at \(T \sim 100\, \text{K}\), for both YbB\(_{12}\) and TmB\(_{12}\). We propose that it is the discovered filamentary structure of the ED that is at the origin of an excitation detected at low temperatures in the terahertz spectra of AC conductivity and permittivity for polarization of the electric field vector \(\mathbf{E}\) of the probing radiation oriented along to the stripes, \(\mathbf{E} \perp [110]\); no such excitation was observed in the case of \(\mathbf{E} \parallel [110]\). Additional evidence is presented by the anisotropy of the magnetoresistance, measured in Tm\(_{1-x}\)Yb\(_x\)B\(_{12}\) for the magnetic field oriented along or across the dynamic stripes. Emergence of stripes in YbB\(_{12}\) is a fundamental finding, indicating exotic dielectric state in the Kondo insulator with metallic Fermi surface.

![Fig. 1](image_url) (a) Crystal structure of RB\(_{12}\) with red and khaki balls marking the RE and boron atoms. Temperature dependences of (b) resistivity \(\rho(T)\) and Hall coefficient \(R_H(T, H \sim 5\, \text{kOe})\) and (c) Hall mobility \(\mu_H(T) = R_H(T)/\rho(T)\) of charge carriers in YbB\(_{12}\) and TmB\(_{12}\) (for comparison). Solid lines in (b) demonstrate the activation behavior of the Hall coefficient. Inset in (c) explains the idea of measuring the angular \(\phi\)-dependence of the magnetoresistance; vector \(n\) is perpendicular to the sample surface. Roman numerals I and II denote the charge transport regimes (see the text).

X-ray data on high-quality YbB\(_{12}\) single crystal (as the same as in Ref.\(^{17}\)) were collected on an Xcalibur diffractometer (Rigaku Oxford Diffraction) equipped with an EOS S2 CCD detector, using Mo \(K_a\) radiation, \(\lambda = 0.71073\, \text{Å}\). The sample cooled using a Cobra Plus cryosystem (Oxford Cryosystems) with an open flow of cold nitrogen directed at the sample (for details, see Ref.\(^{17}\)). A rectangular single-crystalline sample \(4 \times 0.2 \times 0.2\, \text{mm}^3\) in size was prepared from the same batch of YbB\(_{12}\) and used to study the resistivity and Hall resistance in a five-terminal scheme with a direct-current (DC) commutation at temperatures 4.2–300 K in a magnetic field up to 80 kOe. Additionally, the magnetoresistance studies of the single crystals Tm\(_{1-x}\)Yb\(_x\)B\(_{12}\) with \(x < 0.15\) were carried out for comparison. As shown in the inset of Fig. 1(c), the long axis [011] of the sample coincides with the direction of current \(I\), oriented across the external magnetic field \(\mathbf{H}\), and \(\phi\) is the angle between \(\mathbf{H}\) and the [100] axis of the crystal. The measurements of the magnetoresistance were carried out by rotating the crystal around the [011] axis with a step of \(\Delta\phi = 1.8^\circ\). Broad-band terahertz-infrared (frequencies \(\nu \approx 10–1000\, \text{cm}^{-1}\)) spectra of conductivity and dielectric permittivity were obtained by using terahertz time-domain and infrared Fourier-transform spectrometers as discussed in detail in the Supplementary Information.\(^{23}\)

The resistivity \(\rho(T)\) and Hall coefficient \(R_H(T, H = 5\, \text{kOe})\) of YbB\(_{12}\) measured in the range 4.2–300 K are shown in Fig. 1(b), demonstrating the metal-insulator transition upon cooling. For comparison, Fig. 1(b) also presents the charge transport characteristics of metallic TmB\(_{12}\). Both the resistivity and Hall coefficient of YbB\(_{12}\) increase drastically with decreasing temperature. In the two temperature intervals, I (20–300 K) and II (5–12 K), an activation type behavior is evidenced from the analysis of these two charge transport characteristics [see, e.g., solid lines in Fig. 1(b)], assuming two energy scales that correspond to the indirect gap \(E_g/k_B \approx 216\, \text{K}\) and to the intragap excitation \(E_s/k_B \approx 15\, \text{K}\) (\(k_B\) is the Boltzmann constant) estimated from the Hall effect data in accord with the previous results.\(^{1,4,22}\) Detailed analysis of Hall mobility \(\mu_h(T) = R_H(T)/\rho(T)\) will be presented elsewhere. The two regimes I and II are separated in YbB\(_{12}\) by the strong peak at \(T_s \sim 15\, \text{K}\) on the \(\mu_h(T)\) curve [Fig. 1(c)]. Also note that the crossover to the low-temperature coherent regime of charge transport at \(T_s\) was attributed in Ref.\(^{4}\) to emergence of large size-sized clusters of the many-body states in the Yb-based narrow gap semiconductors (also see Ref.\(^{24}\)). Moreover, it is argued\(^{16}\) that the filamentary structure of the conduction electrons is composed of the charge stripes in the RE dodecaborides, and \(T_s\) is the measure of the electron density fluctuations: \(k_B T_s \sim h\nu_s \sim 1\, \text{meV}\) (\(h\) is Planck’s constant).

Crystal structures of Kondo insulator YbB\(_{12}\)\(^{17}\) and metal TmB\(_{12}\)\(^{18}\) were studied earlier in the range 85–293 K based on x-ray diffraction data. Weak static Jahn–Teller distortions of the cubic lattice were detected at all
temperatures,\textsuperscript{[18]} but this did not require a revision of the highly symmetrical structural model. Both structures were refined at all temperatures in the cubic space group \textit{Fm\overline{3}m}. The intensities of diffraction reflections were measured at a temperature of 107 K, and the results of refinement of the structural models of TmB\textsubscript{12} and YbB\textsubscript{12} are used in this work to calculate difference Fourier syntheses of the electron density \(\Delta g\).\textsuperscript{[23]} As seen in Fig. 2, positive stripe-like electron density residues are observed along the [110] direction in both YbB\textsubscript{12} and TmB\textsubscript{12}. The stripes, which are more pronounced in YbB\textsubscript{12}, are observed not only in rows with metal atoms, but also in rows consisting of only B\textsubscript{12} cuboctahedrons. In the case of TmB\textsubscript{12} the ED distribution in stripes is wider and located mainly near the boron atoms. The axes in the crystal indicated in Fig. 2(a) correspond to the directions shown in the presentation of the magnetoresistance data below. It is worth noting here that the linear stripes along the [110] direction were detected in LuB\textsubscript{12},\textsuperscript{[20]} but quite different patterns of fluctuating charges were found for ZrB\textsubscript{12}, where the charge stripes form two grids from rhombohedrons (checkerboard patterns) built from (i) hybridized 4\textit{d}-2\textit{p} and (ii) only 2\textit{p}

\begin{figure}[ht]
\centering
\includegraphics[width=\textwidth]{fig2.png}
\caption{Difference Fourier synthesis of electron density \(\Delta g\) in YbB\textsubscript{12} (a) and TmB\textsubscript{12} (b) crystals studied at temperature 107 K. The distribution \(\Delta g\) in a layer 1-\AA-thick was obtained by summing over parallel (01\overline{1}) sections with steps of 0.05\AA normalized to the layer thickness. The blue circles indicate the boron positions in the layer or near the layer; the light blue and pink circles mark the Yb and Tm positions, respectively. Shades of green and red indicate areas on the map with negative and positive values of \(\Delta g\), respectively. Contour intervals are 0.2 \(e/\AA^3\).}
\end{figure}

\begin{figure}[ht]
\centering
\includegraphics[width=\textwidth]{fig3.png}
\caption{Terahertz (dots) and infrared (lines) spectra of conductivity (a) and of real (b) and imaginary (c) dielectric permittivity of YbB\textsubscript{12} single crystal measured at different temperatures and polarizations as indicated. Solid circles in (a) and dashed lines in (a)–(c) show the results of Kramers–Kronig analysis of reflectivity spectra. Arrows and stars in panels (a) and (c) indicate direct gap and collective mode in the spectra, respectively. Horizontal arrow in panel (a) indicated DC conductivity measured at \(T = 80\) K. Panels (d), (e), and (f) show the terahertz spectra measured at \(T = 3\) K (red and black lines) and \(T = 13\) K (green line) for two different polarizations. Small oscillations at terahertz frequencies (below 100 cm\textsuperscript{-1}) are caused by standing waves due to multiple reflections of the radiation within the crystal.

Figure 3 shows the terahertz-infrared spectra of conductivity as well as real and imaginary permittivity of the YbB\textsubscript{12} single crystal measured at different temperatures in two polarizations with AC electric field oriented in the sample surface plane (111) along and across the [110] axis. The spectra look similar to those measured previously with unpolarized radiation.\textsuperscript{[25]} The conductivity spectra for both polarizations practically coincide at in-
termediate temperatures [see, e.g., the spectrum measured at 80 K in Fig. 3(a)]. Note that, at 80 K, the $\sigma(\nu)$ behavior is typical for conductor: the Drude-type conductivity increases below 100 cm$^{-1}$ toward the DC value. Upon cooling, strong drop in the conductivity spectrum is observed below 300 cm$^{-1}$ (\sim 40 meV) evidencing opening of a direct gap [indicated by vertical arrows in Figs. 3(a) and 3(c)] in the electron density of states (also see Refs. [25,26]). At $T = 10$ K, two more features are distinguished at frequencies below 300 cm$^{-1}$, as seen in the three panels of Fig. 3: (a) a shoulder at about 150 cm$^{-1}$ in $\sigma(\nu)$ and $\varepsilon''(\nu)$ spectra and (b) a peak at 60–70 cm$^{-1}$ for $E \perp [110]$. Note that no similar peak is seen for the case of $E \parallel [110]$.

From Fig. 3(d) one can see that the position of the peak shifts from \sim 72 cm$^{-1}$ at $T = 13$ K to \sim 62 cm$^{-1}$ at $T = 3$ K. We propose that the origin of the peak may be attributed to the plasmonic oscillations of charge carriers that are delocalized within the stripes elongated transverse to the [110] direction and driven by AC electric field of the terahertz radiation. Similar plasmonic excitations have been observed before in experiments on carbon nanotubes (see, e.g., Ref. [27]). Within such an assumption and following the lines used in Ref. [27], we estimate the distance between the charge carriers that are localized, i.e., stripes length, as $L = V_F(\pi \nu_p)^{-1}$, where $\nu_p$ is the position of the plasmon peak and $V_F$ is the plasmon velocity that is several times larger than the Fermi velocity, $V_F \approx \nu_p$. Taking the stripes energy of \sim 1 meV (charge fluctuations with the frequency $\nu_p \sim 240$ GHz$^{-1}$) we estimate the Fermi velocity of the carriers as $V_F \approx 1.9 \times 10^6$ cm/s. With $\nu_p = 72$ cm$^{-1}$ (at 13 K) and $\nu_p = 62$ cm$^{-1}$ (at 3 K) the average stripes length is evaluated to increase from \sim 100 Å at $T = 13$ K to \sim 116 Å at $T = 3$ K. In Ref. [25], a clear maximum in the AC conductivity spectra of YbB$_{12}$ was also observed in an unpolarized radiation, but only at another frequency of 20 cm$^{-1}$. Lower frequency can indicate larger stripes length in the crystal measured in Ref. [25].

As established earlier,$^{[4,18]}$ taking into account the ED distribution estimated from the XRD data in YbB$_{12}$, the anomalies in the angular dependences of the low-temperature magnetoresistance (MR) can be explained by the scattering of charge carriers on the dynamic charge stripes, which were found previously$^{[4,18]}$ both in the non-magnetic reference dodecaboride LuB$_{12}$ and in the paramagnetic state of RB$_{12}$ with trivalent magnetic RE ions ($R = Ho, Er, and Tm$). In more detail, the inhomogeneous distribution of ED (nanoscale phase separation) in RB$_{12}$ ($R = Lu, Ho, Er, and Tm$), induced by the cooperative JT instability of B$_{12}$ clusters, leads to an additional positive component in both the MR$^{[4,29]}$ and the Hall resistivity$^{[30]}$ due to the interaction of the filamentary structure of fluctuating charges with an external magnetic field. According to Refs. [4,29,30], this interaction has a maximum if the magnetic field is transverse to the dynamic stripes, which are directed along the [110] axis in all dodecaborides with $R^{1+}$ ions. Magnetic field and angular dependences of MR $\Delta \rho(\nu) = [\rho(H) - \rho(0)/\rho(0)]$ at $T = 4.2$ K are presented in Fig. 4 for TmB$_{12}$, YbB$_{12}$, and Tm$_{1-x}$Yb$_{x}$B$_{12}$ with $x < 0.15$. As shown in Fig. 4(b), for

![Fig. 4. Magnetic field [(a), (c)] and angular [(b), (d)] dependences of MR $\Delta \rho(\nu) = [\rho(H) - \rho(0)/\rho(0)]$ at $T = 4.2$ K for TmB$_{12}$ and YbB$_{12}$, correspondingly. Each angle $\phi$ corresponds to the crystallographic axis in the (011) plane, which is directed along $H$ when the crystal is rotated through the angle $\phi$ around the [011] axis. Panels (e) and (f) (enlarged scale) demonstrate the MR anisotropy changes at $T = 2$ K and $H = 80$ kOe when the Tm is partially replaced by Yb in Tm$_{1-x}$Yb$_{x}$B$_{12}$ [see $x$(Yb) values on the right of panels (e) and (f)].](127302-4)
The MR reaches its maximum for $H || [100]$, but in YbB$_{12}$ the low-temperature MR anisotropy changes its polarity [Fig. 4(d)]. The sharp change in the MR anisotropy can be explained by the reorientation below $T_s \sim 15$ K of the ED stripes in YbB$_{12}$ from [110] to transverse direction, which is caused by strong 4$f$–5$d$ charge and spin fluctuations at the Yb ions. As a result, we can assume the appearance of new configurations of dynamic charge stripes in YbB$_{12}$ additionally to those caused by the cooperative JT instability of the rigid boron frame. To verify the MR changes in the presence of Yb ions we investigated, for comparison, several Tm$_{1-x}$Yb$_x$B$_{12}$ single crystals with $x < 0.15$. It is seen from Figs. 4(e)–4(f) that the drastic MR variation occurs already in the metallic state in the range of $0.5$ at.% $< x < 0.5$ at.% of Yb in Tm$_{1-x}$Yb$_x$B$_{12}$ arguing in favor of the proposed scenario with transformation in the stripe patterns.

Note that the XRD results in Fig. 2 indicate the [110] direction of the dynamic charge stripes in both YbB$_{12}$ and TmB$_{12}$ above 100 K, at least. Below $T_s \sim 15$ K [to the left of the mobility maximum in Fig. 1(c)], new patterns of the ED filamentary structure are expected to dominate, which are determined mainly by the 4$f$–5$d$ charge fluctuations at the Yb positions, and consequently, the MR anisotropy changes polarity [Figs. 4(b) and 4(d)]. The low temperature transformation occurs even for small Yb contents in Tm$_{1-x}$Yb$_x$B$_{12}$ solid solutions [Figs. 4(e) and 4(f)]

Thereby, low-temperature charge fluctuations become transverse to [110], which leads to appearance of polarization effects in the terahertz conductivity of YbB$_{12}$, where a plasma peak is observed at $60–70$ cm$^{-1}$ for polarization $E \perp [110]$ (Fig. 3). The transformation of YbB$_{12}$ stripe patterns well below $T_d \sim 120$ K correlates very well with dramatic changes above $T_s \sim 15$ K in the spectra of magnetic excitations detected in Ref. [31] by the inelastic neutron scattering (INS). Indeed, it was found [31] that, with increasing temperature, the magnetic excitations start to transform: the spin gap is gradually filled with a broad quasi-elastic signal (half width $\Gamma/2 \sim 10$ meV), while three narrow INS peaks below 40 meV are concurrently suppressed. The transformation to the high-temperature regime of spin fluctuations ends near $T_d \sim \Gamma/2 \sim 120$ K, and the energy of spin (and charge) fluctuations is considered as the boundary between these two regimes in YbB$_{12}$ with an intermediate valence of Yb ions. Similarly to the ED patterns at $T \sim 100$ K (Fig. 2), the low-dimensional character of the spin fluctuation spectrum was observed in YbB$_{12}$ [32] and the temperature dependence of the ESR amplitude was found to be close to exponential increase with a characteristic temperature of $\sim 18$ K, which is close to $T_s \sim 15$ K. The authors [32] concluded that the ESR results can be understood assuming the existence of Yb$^{3+}$–Yb$^{3+}$ ion pairs coupled by isotropic exchange interaction, which also interact with neighboring Yb pairs connecting these dimers in the nanoscale channels. Thus, the chains of Yb pairs in the metallic channels were predicted previously in various independent experiments.

In summary, the dynamic charge stripes have been discovered in the archetypal strongly correlated electron system YbB$_{12}$ with metal-insulator transition by precise XRD data analysis, and confirmed by polarized terahertz spectroscopy and charge transport measurements. At $T \sim 100$ K, near the temperature of spin fluctuations, i.e., $T_d$, YbB$_{12}$ exhibits stripe patterns oriented along the [110] direction, with the corresponding filamentary electron density structure found in the XRD experiments not only between rare-earth metal atoms, but also in rows consisting of B$_{12}$ cuboctahedrons. On the contrary, it is proposed that at liquid helium temperatures $T < T_s \sim 15$ K a crossover to the coherent regime of charge transport takes place, which is accompanied by setting up of the low-dimensional (1D or 2D) spin and charge fluctuations and formation of large size ($>100$ Å) clusters in the nanoscale filamentary structure of conduction electrons oriented transverse the [110] direction within the semiconducting matrix of YbB$_{12}$. We propose the formation of dynamic charge stripes penetrating the semiconducting matrix in YbB$_{12}$ as a fundamental phenomenon, elucidating genesis of exotic dielectric state in the Kondo insulator with metallic Fermi surface.

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