Superconductivity in Strong Spin Orbital Coupling Compound Sb$_2$Se$_3$

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Recently, A$_2$B$_3$ type strong spin orbital coupling compounds such as Bi$_2$Te$_3$, Bi$_2$Se$_3$ and Sb$_2$Te$_3$ were theoretically predicted to be topological insulators and demonstrated through experimental efforts. The counterpart compound Sb$_2$Se$_3$ on the other hand was found to be topological trivial, but further theoretical studies indicated that the pressure might induce Sb$_2$Se$_3$ into a topological nontrivial state. Here, we report on the discovery of superconductivity in Sb$_2$Se$_3$ single crystal induced via pressure. Our experiments indicated that Sb$_2$Se$_3$ became superconductive at high pressures above 10 GPa proceeded by a pressure induced insulator to metal like transition at 3 GPa which should be related to the topological quantum transition. The superconducting transition temperature ($T_C$) increased to around 8.0 K with pressure up to 40 GPa while it keeps ambient structure. High pressure Raman revealed that new modes appeared around 10 GPa and 20 GPa, respectively, which correspond to occurrence of superconductivity and to the change of $T_C$ slope as the function of high pressure in conjunction with the evolutions of structural parameters at high pressures.

One big breaking through in the studies of strong spin orbital coupling (SOC) system is the discovery of A$_2$B$_3$ topological insulators (TIs)$^1$. TIs are characterized by insulating bulk energy gap like in ordinary insulators but gapless edge or surface states protected by time-reversal symmetry, and therefore trigger wide interests in physical sciences$^{1–8}$. As one of the extraordinary states of topological quantum phases, topological superconductor with Majorana bound state at edge or surface, shows a potential application as well as provides theoretical foundation in topological quantum computations$^8$. Superconductivity in TIs was firstly realized in Cu-intercalated Bi$_2$Se$_3$$_{10}$. In this system, the copper atoms reside in the van der Waals gaps between two Bi$_2$Se$_3$ layers resulting in the superconductivity with Cooper pairing existing up to 3.8 K. Further efforts on introducing superconductivity into topological insulator and exploring clean topological superconductors without doping were resorted to high pressure$^{11–13}$. Comparing with chemical substitution, applying pressure is an effective tool of inducing novel properties in topological insulators by directly modifying the electronic structure without introducing defects or impurities. For instance, pressure can effectively tune the crystal field splitting and hybridization between Sb and Se$^{14}$, resulting the emergence of topological insulator state. The A$_2$B$_3$-type TIs (A = Sb, Bi; B = Se, Te) except for Sb$_2$Se$_3$ exhibit rich and novel phenomena under high pressure, such as structure phase transitions$^{11–13,15–21}$, insulator-to-metal transitions$^{13,17}$, and even superconductivity$^{11–13,16–22}$. The bulk superconducting states of Bi$_2$Te$_3$ and Sb$_2$Te$_3$ were observed experimentally as the increase of pressure and were proposed to be likely topologically related$^{11,12}$. Additionally, the high pressure tuned superconductivities were realized in Bi$_2$Se$_3$$_{13,22}$ and Cu$_x$Bi$_2$Te$_3$$_{19}$ system. All these TIs provided possible pathway to search for Majorana fermions through high pressure technique. Sb$_2$Se$_3$ compound, as a member of A$_2$B$_3$-type strong SOC semiconductors (A = Sb, Bi; B = S, Se, Te), has attracted great amount of attention in the past few years, due to its switching effects$^{21}$, high thermoelectric power$^{24}$, and good photovoltaic properties$^{25}$. Unlike Bi$_2$Te$_3$, Bi$_2$Se$_3$ and Sb$_2$Te$_3$, Sb$_2$Se$_3$ was thought to be topological trivial$^1$. However, it was recently theoretically$^{26,27}$ predicted that Sb$_2$Se$_3$ could transform from band insulator to topological insulator under high pressure and thereafter the prediction of topology was experimentally elucidated$^{31}$. Here, we report the effect of pressure on the transporting...
properties of Sb₂Se₃ in combination with structural studies through Raman spectra and in situ angle-dispersive synchrotron x-ray diffraction (AD-XRD). Our experiments indicated that Sb₂Se₃ transformed from insulator into metal like state and further to superconductive state as increase of pressure, during which there was no crystal structure phase transition occurred in our studied pressure range. Two new Raman active modes were observed at high pressure, respectively, where correspondingly the superconductivity emerges and the dTc/dp slope changes.

**Results**

**Electronic transport properties of Sb₂Se₃ single crystal.** Sb₂Se₃ is a band insulator with energy gap of ~1 eV\(^2\) and with resistance at the level of 10\(^9\) Ω at ambient condition. Sb₂Se₃ compound undergoes an insulator to metal like transition upon compression, as shown in Fig. 1 (a). As increasing pressure, the resistance decreases over the entire measured temperature range, showing four orders of magnitude drop at room temperature with pressure up to 3.4 GPa, which could be related to the topological quantum transition\(^2\). The transport must contain conductance contribution from surface but the contribution cannot be differentiated at this moment. From 3.4 to 8.1 GPa, the temperature dependence of resistance is similar to that for topological insulator Bi₂Se₃ with an upturn of resistance at low temperature\(^2\). However, the upturn in resistance is barely visible, due to the high carrier density screening effect\(^2\). In Sb₂Se₃, superconductivity becomes more pronounced with pressure higher than 10 GPa, as shown in Fig. 1 (b). The signature of superconductivity is the resistance drop at around 2.2 K with decreasing temperature under pressure of 10 GPa. Resistance drops more obviously with further increasing pressure and zero-resistance state was fully realized above 15 GPa. Determination of superconducting transition temperature (Tc) was based on the differential of resistance over temperature, as elucidated in Ref. 11. To validate the superconductive character, the evolutions of Tc at 15.4 GPa as a function of magnetic field are performed, as shown in the inset of Fig. 1 (c). The suppression of Tc with increasing magnetic field confirms the superconductivity. Using the Werthamer-Helfand-Hohenberg formula\(^10\), \(H_c^2(0) = \frac{\mu_0}{4\pi}\frac{dH_c(T)}{dT}|_{T=T_c}\), the upper critical field \(H_c^2(0)\) is extrapolated to be 3.9 T with magnetic field \(H\) paralleling to c axis of Sb₂Se₃ single crystal at 15.4 GPa. Figure 2 (a) shows resistance evolution of Sb₂Se₃ compound as the function of pressures at various temperatures. The evolutions of Tc from different experimental runs as functions of pressures are plotted in Fig. 2 (b). Tc increases rapidly to 6 K at 20 GPa with a rate of 0.40 K/GPa. Subsequently, the increase rate of Tc is 0.19 K/GPa and thereafter, Tc increases very slowly to around 8 K at 40 GPa. We further conducted Hall effect measurement with a magnetic field \(H\) perpendicular to the a–b plane of the Sb₂Se₃ single crystal and sweep the \(H\) at fixed temperatures (2 K, 30 K and 297 K, respectively) for each given pressure. The Hall resistance as a function of applied pressure at 30 K in Fig. 1 (d), shows a linear behavior with a positive slope, indicating that Sb₂Se₃ is of hole type carrier over the entire pressure range measured. Carrier densities increase almost two orders of magnitude as pressure increases from 10 GPa to 18 GPa (Fig. 2 (c)). For instance, the carrier

![Figure 1](https://www.nature.com/scientificreports/image/6679.png)
density changes from $10^{19}$ cm$^{-3}$ to $10^{21}$ cm$^{-3}$ with increasing rate of $6.2 \times 10^{19}$ cm$^{-3}$/GPa from 10 GPa to 18 GPa at both 2 K and 30 K, suggesting significant change in the electronic band structure with pressure. Apparently, the rapid increase of $T_C$ of the superconductive phase from 10 GPa to 18 GPa is intimately related to the increase of carrier density, as observed in Bi$_2$Te$_3$, Bi$_2$Se$_3$ or Sb$_2$Te$_3$ systems. The difference is that all these phenomena of Sb$_2$Se$_3$ occur within the initial ambient crystal structure (see Supplementary Fig. S1 online), other than pressure induced crystal structure changes in Bi$_2$Te$_3$, Bi$_2$Se$_3$ or Sb$_2$Te$_3$ compounds. It should be noticed that further increasing pressure will continuously increase the carrier density in the measured pressure range. The upright dotted lines in Fig. 2 represent the pressures, at which insulator to metal like and further to superconducting transitions occur.

Figure 2 gives the pressure induced transitions in Sb$_2$Se$_3$. (a) Resistance evolution of Sb$_2$Se$_3$ single crystal as a function of pressure at fixed temperatures (280 K, 200 K, 2 K), in which insulator, metal like and superconductor are denoted. (b) Pressure dependence of superconductive transition temperature $T_C$ of Sb$_2$Se$_3$. Solid lines are guides to the eye. Increasing rate of $T_C$ decreases upon compression. (c) Pressure induced changes of carrier density at temperature of 2 K, 30 K and 297 K, respectively.

High pressure structural evolution of Sb$_2$Se$_3$ crystal. To understand the pressure induced novel physical properties in terms of structural mechanism, we conducted in situ Raman spectra and angle resolved x-ray diffraction (AD-XRD) measurements at high pressures. The Sb$_2$Se$_3$ crystallizes in space group $Pbnm$ at ambient condition and sketch of the coordination environment around the Sb1 and Sb2 cations is shown in Supplementary Fig. S2 (a) online. The AD-XRD results (see Supplementary Fig. S1 online) reveal that Sb$_2$Se$_3$ maintains ambient-pressure phase within the
pressure range in our experiment, consistent with the recent observation. At this point, the ambient structure of Sb2Se3 stabilizes within entire pressure range we studied, indicating that insulator to metal like to superconducting transitions as increase of pressure is type of electronic phase changes related to local structure evolutions. Raman spectroscopy is sensitive to local bond vibrations and symmetric broken as shown in Supplementary Fig. S2 (b) and (c) online. At ~10 GPa, the sign of increase rate of shear 2-Se3 sharply drop to ~19\(^\circ\) and increases linearly with pressure to ~10 GPa, as shown in Fig. 5 (b). The a/b ratio reaches to 1 at 2.5 GPa and followed by parabolic trend with pressure up to 10 GPa. As mentioned above, the crystal field splitting and hybridization must be modulated accordingly. More specifically, the variation of Mn-Sb-Se3 will mainly rotate the polyhedral of Se(1)Se$_2$ along the c axis while evolution of Mn-Sb-Se3 will tilt and distort the polyhedral of Se(1)Se$_2$ at high pressure, as the schematic of the crystal structure shown in Supplementary Fig. S2 (b) and (c) online. At ~10 GPa, the sign of increase rate of Mn-Sb-Se3 suddenly increases (as shown in Fig. 5 (b)), and a new Raman vibrational mode MM at 200 cm$^{-1}$ emerges (as shown in Fig. 5 (d)), which should correspond to the metal like state to a superconductive state transition in transport measurement. The values of angles of Mn-Sb-Se3 and Mn-Sb-Se3 in Fig. 5 (b) decrease in the pressure range of 10 to 20 GPa with a slope of $-0.425'$/GPa and $-0.295'$/GPa, respectively, then Mn-Sb-Se3 change to a smaller decreasing rate and Mn-Sb-Se3 turns to increase with pressure higher than 20 GPa. At 20 GPa, the change of increase rate of T$_C$ should be due to anomalies of Mn-Sb-Se3 and Mn-Sb-Se3 in Fig. 5 (b) and the appearance of another new Raman vibrational mode MM at ~190 cm$^{-1}$. The dependence of T$_C$ in Sb2Se3 on pressure is similar to that in Bi$_2$Se$_3$, in which pressure-induced unconventional superconducting phase has been reported. From 10 GPa to ~20 GPa, the increased carrier density suggests an increased electronic density of states, which promotes an increase of T$_C$. The a/b ratio stays close to 1 between 10 and 20 GPa followed by a decrease with higher pressure, as shown in Fig. 5 (c). Further increasing pressure to 30 GPa, T$_C$ keeps increasing. Like Bi$_2$Se$_3$, balanced electronic contribution, phonon contribution and some other parameters may lead to the very slow variation from 30 GPa to 40 GPa (see Fig. 2 (b)). However, the relationship between T$_C$ increase (>20 GPa) and carrier density need to be elaborated and established by further experiments.

The complex structure versus rich properties evolution of Sb2Se3 as a function of pressure, such as ET transition and topological superconducting state, invites further studies from theoretical prediction (electronic structural calculation) to validate the possible topological character.
Methods

Sample synthesis. Sb$_2$Se$_3$ single crystal was grown by Bridgeman method. High purity Sb and Se elements are mixed at a molar ratio of 2:3, grounded thoroughly in an agate mortar to ensure homogeneity and then pressed into a cylinder. The mixture was sealed in an evacuated quartz tube under vacuum of 10$^{-2}$ Pa and then heated to 850°C for 24 hours. The quartz tube and sample inside were slowly cooled down to 500°C with a controlled rate of 10°C/h, then followed by furnace cooling to room temperature. The crystal quality was assured by x-ray diffraction & EDX measurements.

Measurement of high pressure properties. The resistance measurement of Sb$_2$Se$_3$ single crystal under pressure was using the standard four-probe method in a diamond anvil cell (DAC) made of CuBe alloy. The diamond was 300 μm in diameter, center flat with 8 degree bevel out to 500 μm. The diamond was 300 μm in diameter, center flat with 8 degree bevel out to 500 μm. A T301 stainless steel gasket was preindented from a thickness of 250 μm to 40 μm, and a hole was drilled at center with diameter of 150 μm. Fine cubic boron nitride (cBN) powder was used to cover the gasket to keep the electrode leads being insulated from the metallic gasket. The cBN powder was pressed and further drilled into a center chamber with a diameter of 100 μm, in which a Sb$_2$Se$_3$ single crystal piece in dimension of 60 μm × 50 μm × 30 μm was loaded simultaneously with soft hexagonal boron nitride (hBN) fine powder surrounding it as pressure transmitting medium. Slim gold wires of 18 μm in diameter were used as electrodes. Pressure was determined by ruby fluorescence method. The DAC was placed inside a MagLab system to perform the experiments with an automatic temperature control. A thermometer located near the diamond of the DAC is used for monitoring the sample temperature. The Hall coefficient at high pressure was measured using the van der Pauw method. The ac susceptibility was measured using inductance method. Two identical coils were wound with one is placed around diamond anvil and the compensating coil directly adjacent. The Sb$_2$Se$_3$ single crystal in dimension of 150 μm × 150 μm × 100 μm was placed into the preindented CuBe gasket chamber with

Figure 4 | Pressure evolution of Raman spectra ($\lambda = 532$ nm, $T = 300$ K). From low to high wavenumber, phonon modes are denoted as M1, M2, M3, M4, M5, M6 and M7 at 0.9 GPa. The black squares are experimental data points, the red solid lines are the total fitting curves from the sum of the individual Lorentzian fits (blue peaks) to the experimental data points. Arrows mark the appearances of new vibration modes.
neon loaded as pressure transmitting medium. Ruby spheres served as a pressure marker.

Raman spectroscopy & angle-dispersive powder x-ray diffraction measurement
sample served as a pressure marker.

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slop as the function of high pressure, and all the solid and dotted lines are

evolutions of Raman vibration modes as functions of pressures. The vertical shadowed boxes indicate the pressures for

Figure 5 | The relation of electrical properties and structure as a function of pressure. (a) $T_c$ from two individual resistance measurement experiments, (b) the angles of $\alpha$-Se1-Sb2-Se3 and $\beta$-Se3-Sb2-Se3, (c) $a/b$ ratio and (d) evolutions of Raman vibration modes as functions of pressure. The monochromatic ADXRD measurements at pressure were performed at the 16BMD beamline of the High Pressure Collaborative Access Team (HCAT) at Advanced Photon Source (APS) of Argonne National Lab (ANL), using a symmetric DAC. The diamond culet was 300 $\mu$m in diameter. The hole was compressed to 120 $\mu$m in diameter. Ruby spheres were pressure

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Acknowledgments
This work was supported by NSF & MOST of China through research projects. C.Q.J. acknowledges H.W.Weng, X.Dai & Z.Fang for discussions. W.G.Y, Y.Z.Y, H.K.M. acknowledge support by EFere, an Energy Frontier Research Center funded by the U.S. Department of Energy (DOE) under Award DE-SC0001057. HP1CAT operations are supported by DOE-NNSA under Award No. DE-NA0001974 and DOE-BES under Award
No. DE-FG02-99ER45775, with partial instrumentation funding by NSF. APS is supported by DOE-BES, under Contract No. DE-AC02-06CH11357.

Authors contributions
C.Q.J. conceived & coordinated the work; L.Y.X. grown single crystals with preliminary characterizations; P.P.K., J.Z., S.J.Z., S.M.F., W.M.L., X.C.W., Q.Q.L. and R.C.Y. conducted the high pressure transport measurements; F.S., X.H.Y., W.G.Y. contributed to the measurements of high pressure structures with the help of Y.S.Z., G.Y.S., H.K.M. and R.A. contributed to the theoretical analysis with help of J.L.Z., C.Q.J., P.P.K. and J.L.Z. analyzed the data; C.Q.J., P.P.K. and J.L.Z. wrote the paper. All authors contributed to the discussions of the work.

Additional information
Supplementary Information accompanies this paper at http://www.nature.com/scientificreports

Competing financial interests: The authors declare no competing financial interests.

How to cite this article: Kong, P.P. et al. Superconductivity in Strong Spin Orbital Coupling Compound Sb2Se3. Sci. Rep. 4, 6679; DOI:10.1038/srep06679 (2014).

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