Supplementary Information

Structural evolution from $Bi_{4.2}K_{0.8}Fe_2O_{9+\delta}$ nanobelts to $BiFeO_3$ nanochains in vacuum and their multiferroic properties

Sining Dong[1,2], *Dalong Zhang*[1], *Yukuai Liu*[1], *Shengwei Yang*[1], *Tao Jiang*[1], *Yuewei Yin*[1] *and Xiaoguang Li*[1,*]

[1]Hefei National Laboratory for Physical Sciences at Microscale, Department of Physics, CAS Key Lab Mat Energy Convers, University of Science and Technology of China, Hefei 230026, P. R. China

[2]Department of Physics, University of Notre Dame, Indiana 46556, USA

* E-mail: lixg@ustc.edu.cn

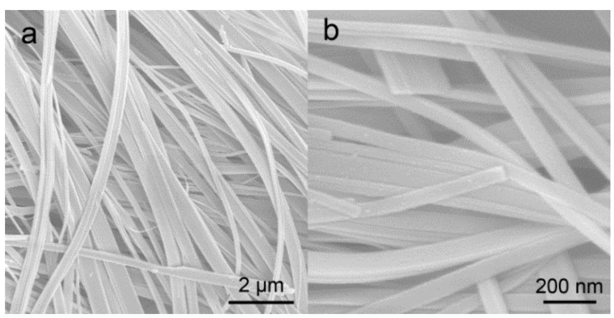

Figure S1. FE-SEM images of the BKFO nanobelts with different magnifications.

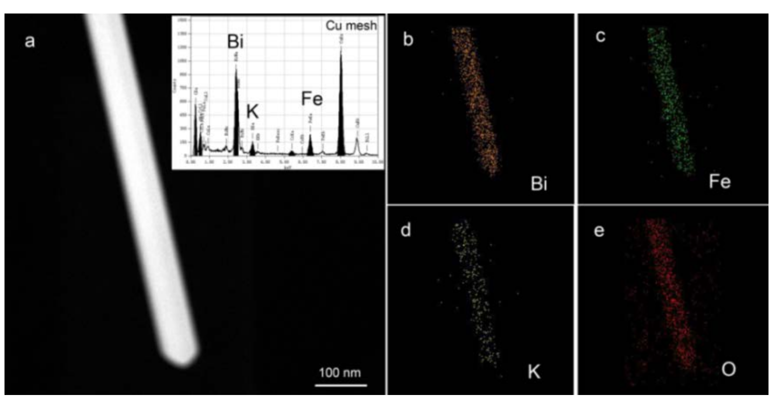

Figure S2. HAADF image (a) and EDX elemental maps (b–e) of a single BKFO nanobelt. Inset of (a) is the EDX spectrum.

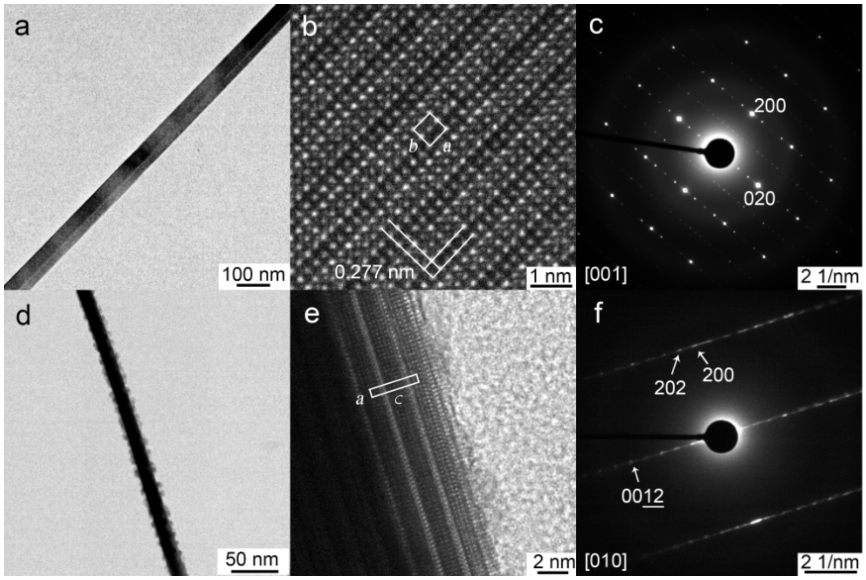

Figure S3. Typical TEM images, HR-TEM images and SAED patterns of $Bi_{4.2}K_{0.8}Fe_2O_{9+\delta}$ nanobelts for the [001] zone axis (a–c) and [010] zone axis (d–f).

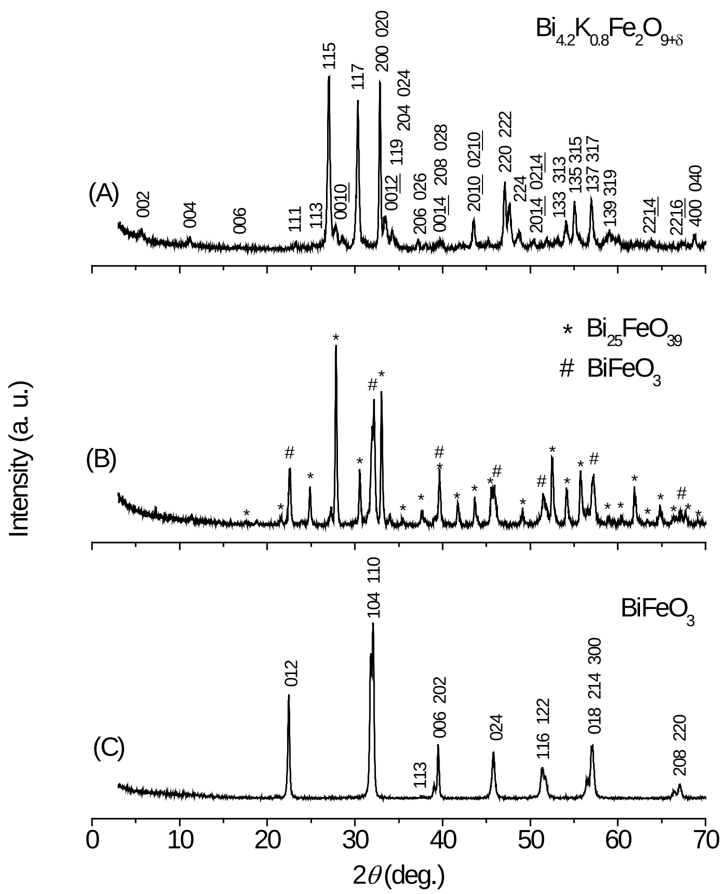

Figure S4. XRD patterns of as-prepared BKFO nanobelts (A), powders after 500 °C vacuum annealing for 2 hours (B), and BFO nanochains (C).

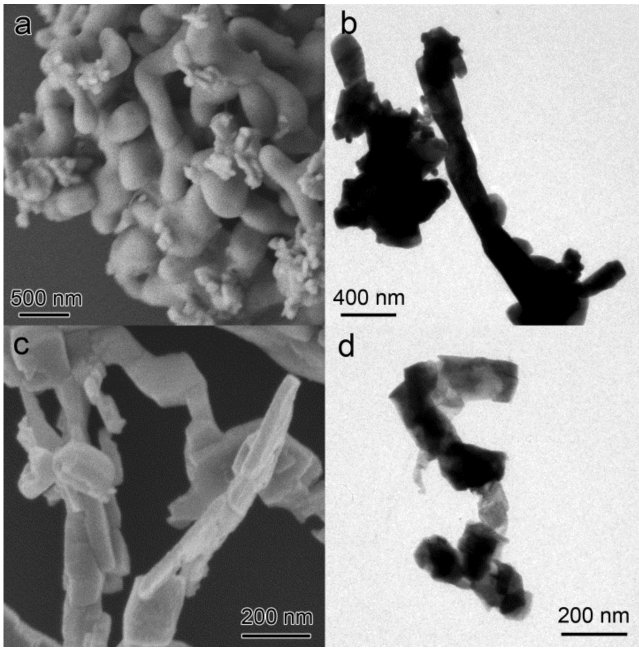

Figure S5. FE-SEM and TEM images of vacuum annealed samples (a, b) and BFO nanochains obtained after cleaning in dilute nitric acid solution (c, d).

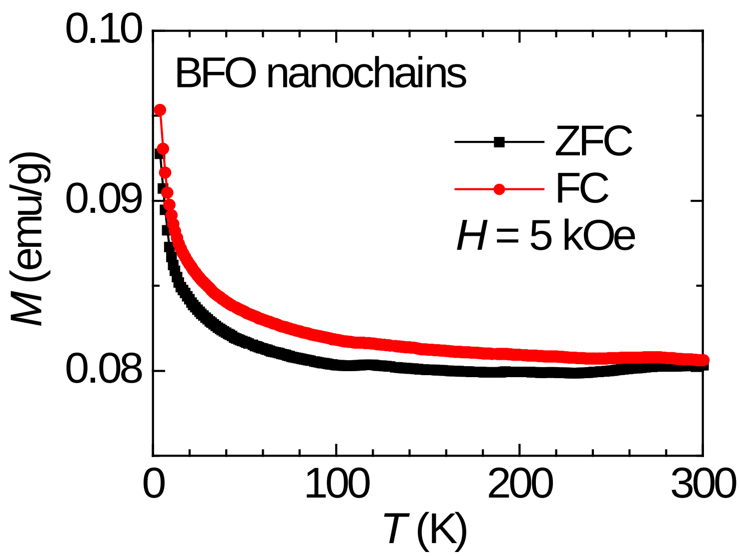

Figure S6. Magnetization of BFO nanochains as a function of temperature for zero-field-cooled (ZFC) and field-cooled (FC) modes measured in a magnetic field of 5 kOe.

Table S1. EDX spectrum analysis results of the atomic percent for $Bi_{4.2\pm0.60}K_{0.8\pm0.12}Fe_2O_{9+\delta}$ nanobelts.*

Samples	Atomic% O K_Series	Atomic% K K_Series	Atomic% Fe K_Series	Atomic% Bi M_Series	Samples	Atomic% O K_Series	Atomic% K K_Series	Atomic% Fe K_Series	Atomic% Bi M_Series
Sample A					**Sample G**				
Spectrum1	67.14762	3.7202	9.47967	19.65251	Spectrum1	68.89456	3.65575	8.66631	18.78338
Spectrum2	67.36723	4.33542	8.73441	19.56293	Spectrum2	77.67585	2.86015	5.90012	13.56388
Spectrum3	72.30125	3.49141	7.95648	16.25086	Spectrum3	76.40348	3.05695	6.7798	13.75977
Spectrum4	73.33473	3.18178	7.69884	15.78465	Spectrum4	72.60213	3.24275	7.33779	16.81732
Spectrum5	43.32582	6.09364	17.98173	32.59881	Spectrum5	75.37449	3.00363	6.68987	14.93202
Sample B					**Sample H**				
Spectrum1	76.38185	2.90094	6.47274	14.24446	Spectrum1	67.67336	3.79213	9.81376	18.72074
Spectrum2	78.1685	2.70433	6.11341	13.01375	Spectrum2	77.2296	3.02211	6.62492	13.12337
Spectrum3	74.46482	3.291	6.80556	15.43863	Spectrum3	70.32492	2.88538	10.61115	16.17855
Spectrum4	73.43842	3.40029	7.42629	15.735	**Sample I**				
Spectrum5	79.83573	2.14934	4.45139	13.56353	Spectrum1	76.173	3.07336	6.85564	13.89799
Sample C					Spectrum2	74.45949	2.49331	5.84728	17.19992
Spectrum1	74.52113	3.02659	7.24842	15.20386	Spectrum3	78.33902	2.88491	6.01524	12.76083
Spectrum2	63.67762	4.07554	11.35529	20.89155	**Sample J**				
Spectrum3	68.86365	3.49373	8.70824	18.93438	Spectrum1	75.46947	2.95378	6.79683	14.77992
Spectrum4	73.51319	2.38528	6.19255	17.90898	Spectrum2	70.02698	2.8256	7.08818	20.05924
Spectrum5	78.80101	2.57233	5.85329	12.77338	Spectrum3	80.18475	2.44554	5.31813	12.05158
Sample D					Spectrum4	71.9543	3.4519	7.4729	17.1209
Spectrum1	76.11416	3.30555	6.5028	14.07749	**Sample K**				
Spectrum2	67.21686	3.54087	9.64403	19.59824	Spectrum1	71.32803	3.40428	8.02902	17.23867
Spectrum3	78.65385	2.47407	6.17385	12.69822	Spectrum2	68.84116	3.8148	8.8849	18.45914
Spectrum4	74.27336	3.28186	7.10985	15.33494	Spectrum3	76.56029	2.82632	6.45537	14.15802
Spectrum5	76.3706	2.32812	6.84575	14.45553	Spectrum4	74.00261	2.98409	7.5385	15.4748
Sample E					Spectrum5	76.68104	2.80681	6.68913	13.82302
Spectrum1	62.92453	4.23501	10.89161	21.94885	**Sample L**				
Spectrum2	71.42111	3.49587	8.22434	16.85869	Spectrum1	73.68294	3.34994	7.19969	15.76743
Spectrum3	71.96433	3.42613	8.01243	16.59711	Spectrum2	77.43375	2.29315	4.83838	15.43471
Spectrum4	76.33911	3.26822	6.6032	13.78946	Spectrum3	78.10831	2.90855	5.93599	13.04715
Spectrum5	68.49602	3.69909	9.27094	18.53395	**Sample M**				
Sample F					Spectrum1	74.82156	3.29134	6.9399	14.9472
Spectrum1	71.28347	3.83251	8.73569	16.14833	Spectrum2	69.30002	3.84382	8.94192	17.91423
Spectrum2	73.23496	3.17102	7.64272	15.95131	Spectrum3	69.19326	3.52103	8.87839	18.40732
Spectrum3	76.85841	2.66708	5.72892	14.74559	Spectrum4	73.14534	3.52619	7.49289	15.83558
Spectrum4	73.269	3.06378	7.86735	15.79987	Spectrum5	75.42998	3.04581	6.15561	15.3686
Spectrum5	59.62489	4.12255	12.78345	23.4691	Spectrum6	72.64045	3.3528	8.12984	15.87691

Table S1. (Continued)

Samples	Atomic% O K_Series	Atomic% K K_Series	Atomic% Fe K_Series	Atomic% Bi M_Series	Samples	Atomic% O K_Series	Atomic% K K_Series	Atomic% Fe K_Series	Atomic% Bi M_Series
Sample N					**Sample S**				
Spectrum1	72.39411	2.89284	7.96372	16.74933	Spectrum1	73.83192	3.28054	7.09963	15.78791
Spectrum2	68.6136	3.55519	9.05238	18.77884	Spectrum2	67.92727	3.5374	8.93763	19.59771
Spectrum3	73.05707	3.47256	7.45437	16.016	Spectrum3	72.10946	3.54277	7.86123	16.48654
Spectrum4	76.16168	2.94414	6.91027	13.98391	Spectrum4	68.89889	3.44809	9.25201	18.40101
Spectrum5	76.18575	2.75598	6.23261	14.82566	Spectrum5	63.55811	3.57671	11.50362	21.36156
Sample O					**Sample T**				
Spectrum1	75.57629	3.03806	6.74345	14.64219	Spectrum1	74.61641	2.75261	6.43652	16.19447
Spectrum2	73.00775	3.40641	7.63301	15.95283	Spectrum2	75.00163	3.106	6.68879	15.20357
Spectrum3	60.90742	3.007	16.04631	20.03928	Spectrum3	77.70314	2.8974	6.19567	13.2038
Spectrum4	76.12058	2.65942	6.1599	15.0601	Spectrum4	78.99588	2.49325	6.05176	12.45911
Spectrum5	77.79806	2.32613	4.75095	15.12487	Spectrum5	70.17872	3.70249	7.90927	18.20952
Sample P					Spectrum6	74.21363	3.21902	7.07425	15.49309
Spectrum1	64.10566	3.75456	10.95336	21.18642	**Sample U**				
Spectrum2	78.10294	2.48397	7.00942	12.40367	Spectrum1	75.88947	2.97647	7.01204	14.12202
Spectrum3	77.96607	2.80778	6.99969	12.22646	Spectrum2	76.01575	2.93389	7.41888	13.63149
Spectrum4	78.14964	2.89397	6.29838	12.65802	Spectrum3	76.80831	2.92014	6.75885	13.5127
Spectrum5	67.80204	3.52051	10.01459	18.66286	Spectrum4	76.82463	3.15475	6.16587	13.85475
Sample Q					Spectrum5	76.13268	3.08131	6.33638	14.44963
Spectrum1	73.25785	3.14968	7.69777	15.89471	Spectrum6	78.18756	2.56486	6.08207	13.16551
Spectrum2	64.4161	3.73795	10.56503	21.28092	**Sample V**				
Spectrum3	63.88316	4.09469	11.28985	20.7323	Spectrum1	79.09938	2.67194	5.98887	12.23981
Spectrum4	75.17301	3.24677	7.16985	14.41036	Spectrum2	72.00544	3.11214	7.70979	17.17262
Spectrum5	64.53414	4.11582	11.9677	19.38234	Spectrum3	73.41149	3.41302	7.52027	15.65522
Sample R					Spectrum4	59.3329	4.75895	12.00114	23.90702
Spectrum1	70.47198	3.45855	8.25168	17.81779	Spectrum5	60.21961	4.12946	13.15246	22.49847
Spectrum2	73.6297	3.49491	7.53681	15.33858	**Sample W**				
Spectrum3	57.38057	5.07507	12.97863	24.56573	Spectrum1	68.97516	3.98884	9.0812	17.95479
Spectrum4	52.98713	4.78748	14.71737	27.50802	Spectrum2	71.54544	3.63658	7.93939	16.87859
Spectrum5	55.72513	4.43579	14.21034	25.62874	Spectrum3	65.20673	3.42065	8.52887	22.84375
Spectrum6	50.27627	3.30946	20.65142	25.76285	Spectrum4	60.28223	4.09564	10.88405	24.73809
					Spectrum5	63.58463	3.74687	10.62995	22.03855

***** For these EDX measurements, there is a considerable oxygen background in the instrument system, and the layered structure of BKFO could also adsorb extra oxygen, so the atomic percentages of oxygen are not the real oxygen contents in the sample. However, we can obtain a relative precise atomic ratio for Bi, K, and Fe. If we take Fe as the benchmark, Bi : K : Fe = 4.2(±0.60) : 0.8(±0.12) : 2. The atomic percent of oxygen in BKFO can be finally estimated through the valence balance analysis as well as the structure refinement. Then, the chemical formula for the BKFO nanobelts can be expressed as $Bi_{4.2\pm0.60}K_{0.8\pm0.12}Fe_2O_{9+\delta}$.

Captions of the Supplementary Videos

SVideo 1. The appearance and the formation of Bi nanoparticles on a $Bi_{4.2}K_{0.8}Fe_2O_{9+\delta}$ nanobelt.

SVideo 2. Ultra-fast fusion of two 3 nm Bi nanoparticles on the $Bi_{4.2}K_{0.8}Fe_2O_{9+\delta}$ nanobelt surface.

SVideo 3. Slow fusion and crystallization of 10 nm Bi nanoparticles on the surface of a $Bi_{4.2}K_{0.8}Fe_2O_{9+\delta}$ nanobelt.

SVideo 4. Dynamic SAED patterns of the $Bi_{4.2}K_{0.8}Fe_2O_{9+\delta}$ nanobelt with Bi nanoparticles on it. The Bi (012) face diffraction points move in a circle due to the rotation movement of the Bi nanoparticles on the nanobelt.