EXPLORING THE CRYSTAL STRUCTURE OF HIGH TEMPERATURE SUPERCONDUCTORS:

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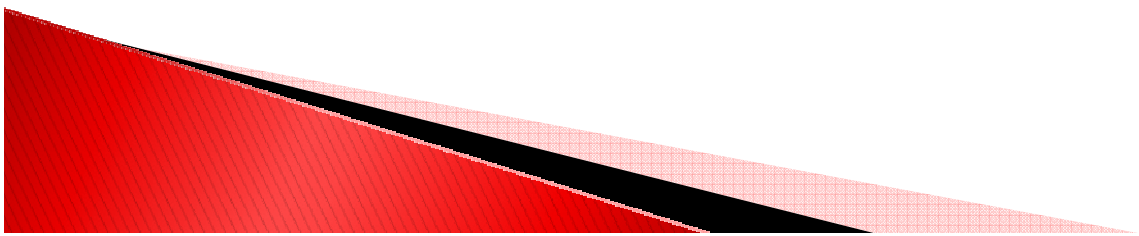
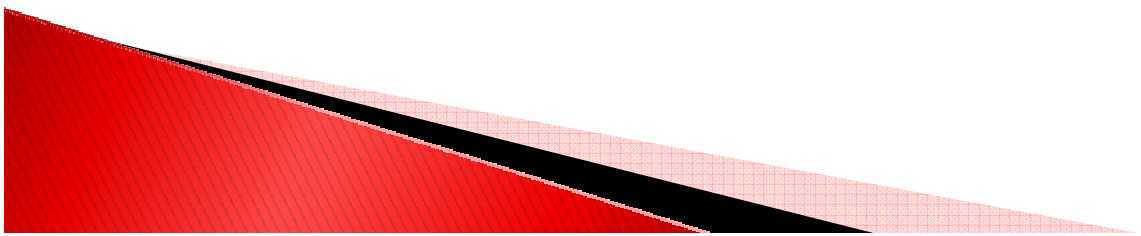
~ THANK YOU



WHAT IS SUPERCONDUCTIVITY?

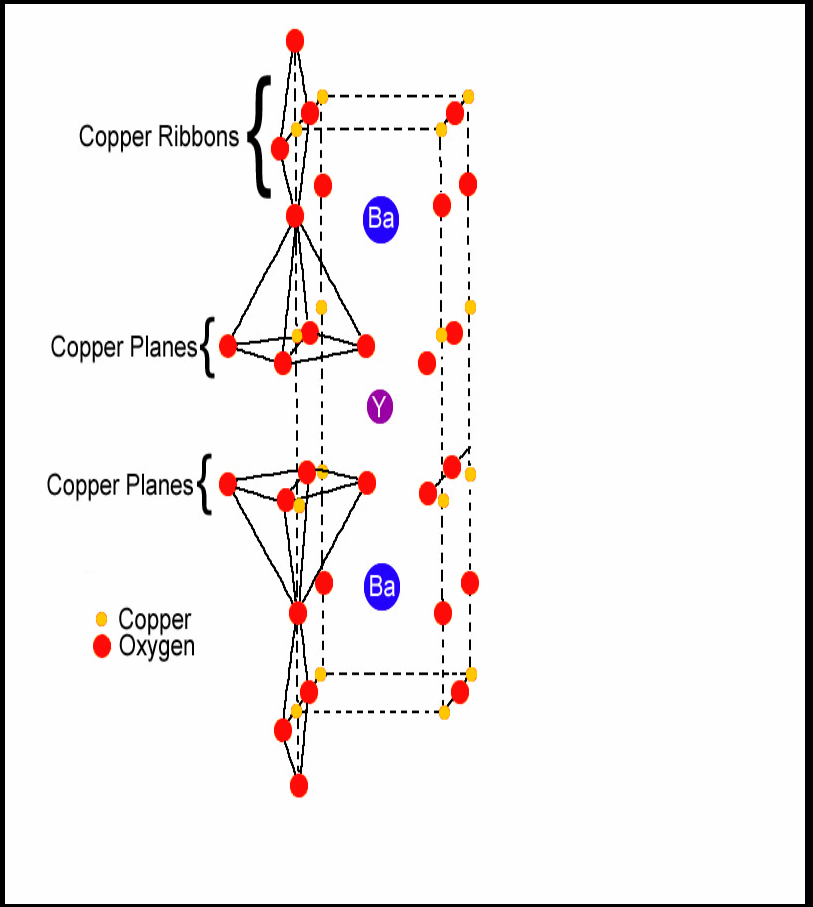
- Superconductivity is the loss of all resistance to the passage of an electric current in certain metals and alloys at low temperatures.

- In some of the new classes of ceramic compounds such as $YBa_2Cu_3O_{6+x}$ (YBCO), superconductivity occurs at significantly higher temperatures.



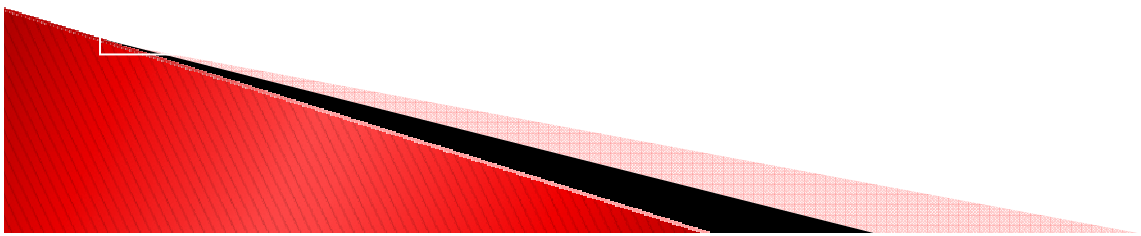
THE STUCTURE OF YBCO:

YBCO crystallizes in a distorted, oxygen-deficient, multi-layered, perovskite structure. The boundary of each layer is defined by planes of square planar CuO_4 units sharing 4 vertices. The planes can some times be slightly puckered. Perpendicular to these CuO_2 planes is CuO_4 ribbons sharing 2 vertices. The yttrium atoms are found between the CuO_2 planes, while the barium atoms are found between the CuO_4 ribbons and the CuO_2 planes.



APPLICATIONS OF YBCO:

- Superconducting Quantum Interference Devices (SQUIDs)
- Magnet Shielding
- Nuclear Magnetic Resonance (NMR)
- Magnetic Levitation Trains
- Power Generation
- Sensors
- Transmission
- Energy Storage



X-RAY DIFFRACTION (XRD):

X-ray diffraction (XRD) is the scattering of x-rays as they interact with the periodic atomic structure of solid matter. Each crystalline compound has a distinct x-ray pattern. The role of x-rays in diffraction experiments is based on the electromagnetic properties of this form of radiation. X-ray can be considered a light but with a very short wavelength. The wavelength is comparable to the inter-atomic distance producing a diffraction pattern by interference of scattered waves.

XRD TECHNIQUES:

There are two main techniques used for finding the diffraction patterns of different materials. These techniques are:

SINGLE-CRYSTAL METHODS:

- This is a method in which an x-ray beam is focused on a single crystal.
- The primary application of this method is to determine the atomic structure, symmetry, unit cell dimensions, etc.

POWDER METHODS:

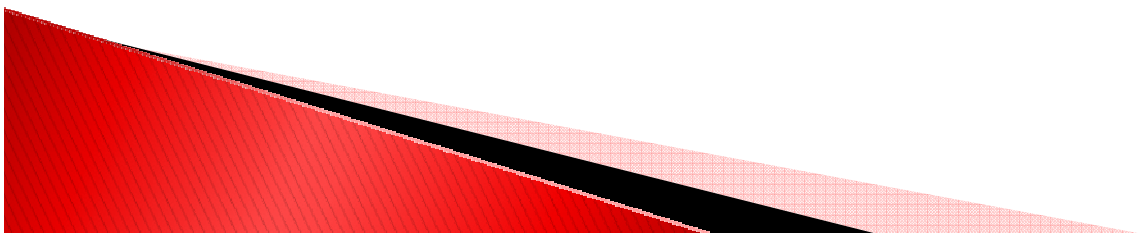
- This is a method in which an x-ray beam is focused on a powder sample composed of small particles. This method is essential for materials that do not form large crystals and it also eliminates the problem of the precise orientation that is needed in the single-methods.
- The primary application of this method is for mineral identification. It can also be used to determine mineral composition along with the relative proportions of minerals in a mixture.

PROS of XRD:

- The significant penetration ability
- During preparation, the creation of thin sections is often avoidable
- A quick and easy way to observe and characterized known, as well as unknown materials

CONS of XRD:

- The inability to provide real-space images of the material under investigation
- The inability to provide quantitative compositional data obtained by the electron microprobe or the textural and quantitative compositional data obtained by the scanning electron microscope



DIFFRACTOMETER:

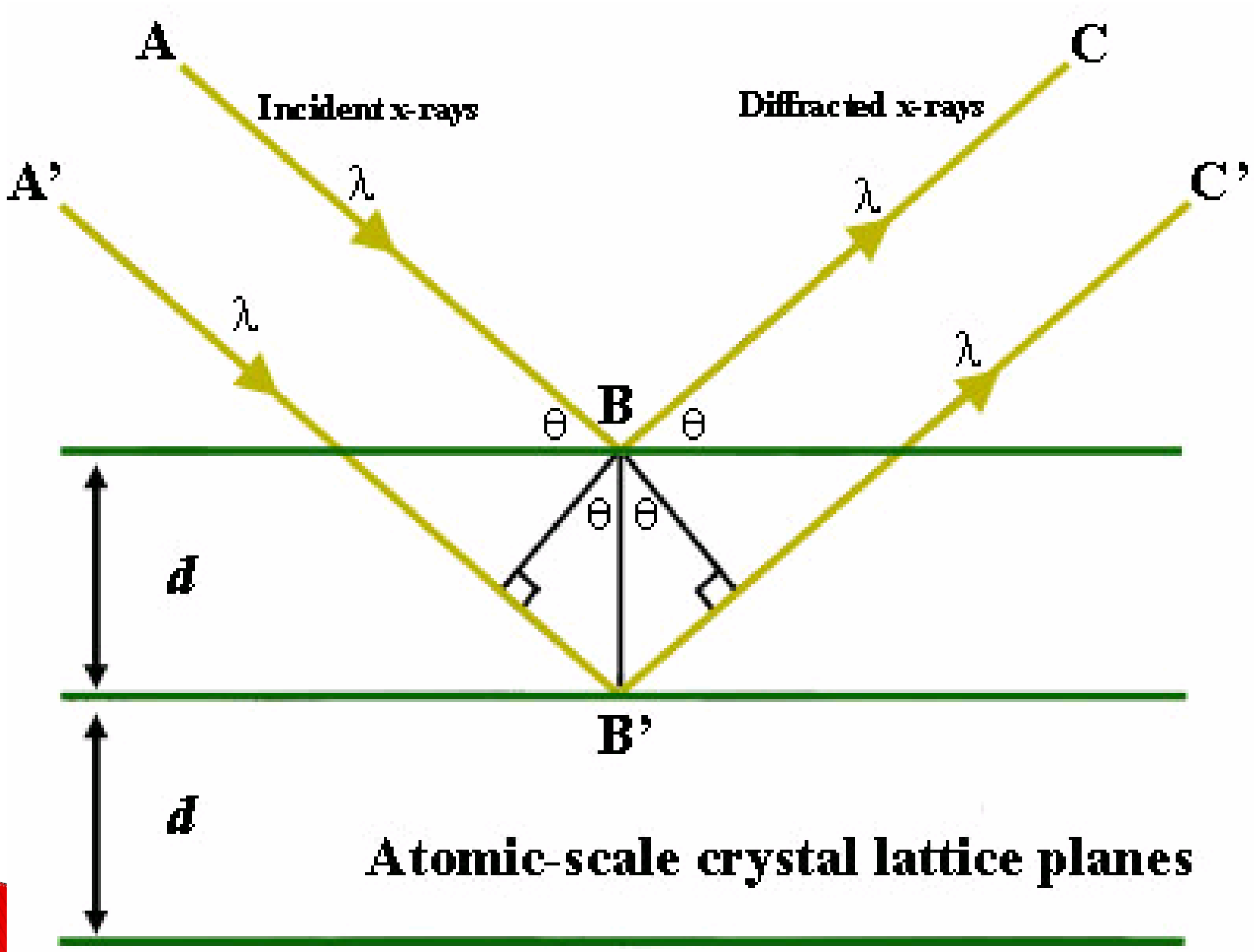
- A diffractometer is a measuring instrument used to make a diffraction pattern of any crystalline solid. With a diffraction pattern an observer can identify an unknown mineral, or characterize the atomic-scale structure of an already identified mineral. The typical diffractometer consists of a source of radiation, a monochromator to choose the wavelength, slits to adjust the shape of the beam, a sample and a detector.

BRAGG'S LAW:

It was derived by the English physicists Sir W. H. Bragg and his son Sir W. L. Bragg in 1913 to explain why the cleavage faces of crystals appear to reflect x-ray beams at certain angles of incidence (theta, Θ). They found that substances whose macroscopic forms were crystalline, gave characteristic patterns of reflected X-radiation. These patterns are unlike those produced by liquids. This observation is an example of x-ray wave interference, or XRD. It was direct evidence of the periodic atomic structure of crystals. The Braggs were awarded the Nobel Prize in Physics in 1915 for their work in determining crystal structures.

When certain geometric requirements are met, x-rays scattered from a crystalline solid can constructively interfere, producing a diffracted beam. Bragg recognized a predictable relationship among several factors. These factors include:

- The distance between similar atomic planes in a mineral, or the d-spacing and measure in angstroms
- The angle of diffraction which we call the theta angle and measure in degrees (Because the diffractometer measures an angle twice that of the theta angle, we call the measured angle 2Θ)
- The wavelength of the incident x-radiation (Lambda)


A
Incident x-rays
C
Diffracted x-rays
A'
C'
λ
λ
λ
λ
B
θ
θ
θ
θ
d
B'
d
Atomic-scale crystal lattice planes

YBCO PELLET PREPERATION:

- First, you must obtain the correct ratio measurements of your starting ingredients, (Yttrium Oxide (Y_2O_3), Barium Carbonate ($BaCO_3$), and Copper Oxide (CuO)) in grams.
- Grind and mix all ingredients together gradually with a mortar and pestle. Do this for about 45 min. or until mixture is a uniform gray color.
- Heat mixture in alumina boat at a temp. of 950 °C for about 16 hours. (Grind and heat – repeat 2 more times, 3 time in all).
- When mixture is completely black it can then be pressed into a pellet.

PROS of SOLID PHASE METHOD:

- This method allows the material to be heated to the required temperature of 950 °C

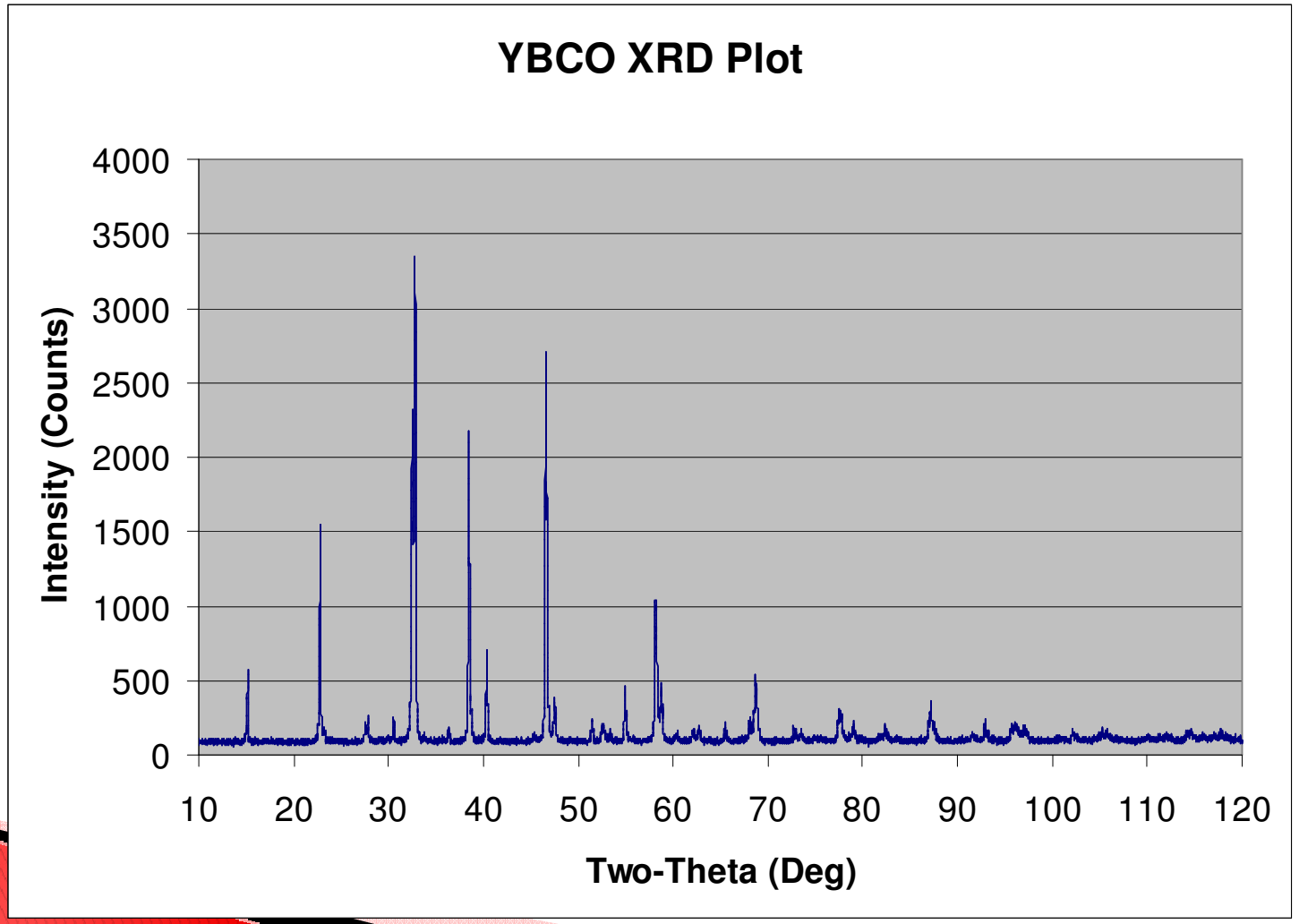
CONS of SOLID PHASE METHOD:

- Large particle size
- The lack of reproductively
- The need for long heat treatment

COLLECTION OF XRD DATA:

- XRD tests are run on YBCO sample
- A print out of a raw data report is provided, then organized
- HKL measurements are determined
- C Language Programming is utilized and an output of data is generate
- Collected data is made into an graph displaying XRD pattern
- All data is combined into a chart, including all experimental and calculated data

X-RAY DIFFRACTION PATTERN OF YBCO:



2-Theta	d(A)	BG	Height	I%	Area	I%	FWHM
15.138	5.8478	82	489	15.1	7377	7.2	0.128
22.794	3.8981	91	1454	44.8	25864	25.4	0.151
23.184	3.8334	96	94	2.9	2915	2.9	0.264
27.563	3.2335	90	126	3.9	3788	3.7	0.256
27.822	3.204	92	171	5.3	5030	4.9	0.25
29.91	2.9849	97	39	1.2	347	0.3	0.076
30.558	2.9231	102	147	4.5	1440	1.4	0.083
32.533	2.75	101	2223	68.6	57011	55.9	0.218
32.783	2.7296	105	3242	100	101901	100	0.267
33.704	2.657	100	52	1.6	615	0.6	0.101
36.331	2.4707	93	91	2.8	1422	1.4	0.133
38.478	2.3376	95	2086	64.3	39978	39.2	0.163
40.341	2.2339	93	612	18.9	11078	10.9	0.154
45.415	1.9954	99	52	1.6	1069	1	0.175
46.6	1.9474	122	2584	79.7	42448	41.7	0.14
47.519	1.9118	124	260	8	5766	5.7	0.189
51.463	1.7742	96	145	4.5	2786	2.7	0.163
52.68	1.736	98	100	3.1	4251	4.2	0.361
54.953	1.6695	97	365	11.3	7486	7.3	0.174
58.189	1.5841	105	938	28.9	30333	29.8	0.275
58.754	1.5702	107	380	11.7	12700	12.5	0.284
60.407	1.5312	98	72	2.2	1645	1.6	0.194
62.07	1.4941	97	71	2.2	2847	2.8	0.341
62.74	1.4797	94	88	2.7	3328	3.3	0.321
65.501	1.4239	92	124	3.8	2563	2.5	0.176
68.741	1.3644	99	424	13.1	17948	17.6	0.36
72.749	1.2988	105	99	3.1	1248	1.2	0.107
72.945	1.2958	101	76	2.3	2273	2.2	0.254
73.492	1.2875	99	75	2.3	2143	2.1	0.243
74.933	1.2663	98	40	1.2	958	0.9	0.204
77.569	1.2297	102	205	6.3	8955	8.8	0.371
77.76	1.2272	101	174	5.4	9306	9.1	0.455
78.991	1.2111	104	133	4.1	3250	3.2	0.208
82.411	1.1693	98	112	3.5	4362	4.3	0.331
87.188	1.1171	97	263	8.1	11289	11.1	0.365
91.59	1.0745	99	58	1.8	1969	1.9	0.289
92.892	1.0629	103	143	4.4	3080	3	0.183
95.738	1.0387	120	94	2.9	2702	2.7	0.244
96.106	1.0357	123	93	2.9	4530	4.4	0.414
97.11	1.0276	136	66	2	1460	1.4	0.188
102.136	0.9902	101	71	2.2	2242	2.2	0.268
105.264	0.9692	107	80	2.5	3344	3.3	0.355
105.79	0.9658	114	57	1.8	1491	1.5	0.222
114.25	0.9172	104	64	2	3372	3.3	0.448
114.609	0.9153	103	74	2.3	3875	3.8	0.445
117.842	0.8994	108	56	1.7	2219	2.2	0.337

CHART OF COMBINED EXPERIMENTAL AND CALCULATED DATA:

H	K	L	I (%)	2-THETA EXP.	2-THETA CAL.	d (A) EXP.	d (A) CAL.	% ERROR
0	0	2	15.1	15.138	15.15823	5.8478	5.8401	0.131847057
0	0	3	44.8	22.794	22.82166	3.8981	3.893399	0.120742827
1	0	0	2.9	23.184	23.24957	3.8334	3.8227	0.279906872
0	1	2	3.9	27.563	27.54175	3.2335	3.235931	-0.075125211
1	0	2	5.3	27.822	27.8711	3.204	3.198436	0.173960023
*	*	*	1.2	29.91		2.9849		
*	*	*	4.5	30.558		2.9231		
0	1	3	68.6	32.533	32.52219	2.75	2.750855	-0.031081246
1	0	3	100	32.783	32.8059	2.7296	2.72771	0.0006928
1	1	0			32.83232		2.725575	0.147675261
1	1	2	2.8	36.331	36.34449	2.4707	2.469839	0.035224968
0	0	5	64.3	38.478		2.3376		
1	1	3	18.9	40.341	40.36116	2.2339	2.232826	0.048100479
*	*	*	1.6	45.415		1.9954		
0	2	0	79.7	46.6	46.69618	1.9474	1.9436	0.19551348
0	0	6						
2	0	0	8	47.419	47.53212	1.9118	1.91135	0.023543569
1	1	5	4.5	51.463	50.17953	1.7742	1.816537	-2.330643417
2	0	2						
1	0	6	3.1	52.68	52.58502	1.736	1.738962	-0.170331497
1	2	0			52.79554		1.732523	0.200689976
1	2	2	11.3	54.953	55.25895	1.6695	1.660975	0.513252758
1	1	6	28.9	58.189	58.23897	1.5841	1.582879	0.077137924
1	2	3						
2	1	3	11.7	58.754	58.77768	1.5702	1.569649	0.035103389
*	*	*	2.2	60.407		1.5312		
*	*	*	2.2	62.07		1.4941		
*	*	*	2.7	62.74		1.4797		
*	*	*	3.8	65.501		1.4239		
2	2	0	13.1	68.741	68.83569	1.3644	1.362788	0.118286924
*	*	*	3.1	72.749	73.57498	1.2988	1.286268	0.974291516
2	2	3						
0	3	0	2.3	72.945	72.95034	1.2958	1.295733	0.005170818
0	3	1	2.3	73.492	73.4709	1.2875	1.287833	-0.02585739
3	0	1	1.2	74.933	74.90326	1.2663	1.266718	-0.025072278
0	3	3	6.3	77.569	77.58934	1.2297	1.229436	0.02196129
1	3	0	5.4	77.76	77.76072	1.2272	1.227154	0.003747332
3	0	3	4.1	78.991	78.99677	1.2111	1.211025	0.006193101
*	*	*	3.5	82.411	83.55029	1.1693	1.156214	1.131797401
3	1	3						
*	*	*	8.1	87.188		1.1171		
2	3	0	1.8	91.59	91.81214	1.0745	1.072515	0.185078997
3	2	0	4.4	92.892	92.57834	1.0629	1.065635	-0.256654483
3	2	1			93.07731		1.061228	0.157553325
*	*	*	2.9	95.738	94.5758	1.0387	1.048326	-0.918225819

CALCULATIONS:

d_{002} = 5.8478 = c/2→ c1 = 11.6956 Å

d_{005} = 2.2339 = c /5→ c2 = 11.1695 Å (outlier)

d_{006} = 1.9474 = c /6→ c3 = 11.6844 Å

d_{003} = 3.8981 = c/3→ c4 = 11.6943Å

C Average = 11.69143333 Å

To measure the dispersion of a set of values the method of standard deviation we used.

STANDARD DEVIATION FORMULA:

$$\sigma = \sqrt{\frac{1}{N}\sum_{i=1}^{N}(x_i - \overline{x})^2}$$

We applied this formula to each of the c values we calculated. When we were done, these were the values we obtained.

STANDARD DEVIATION:

Original numbers:

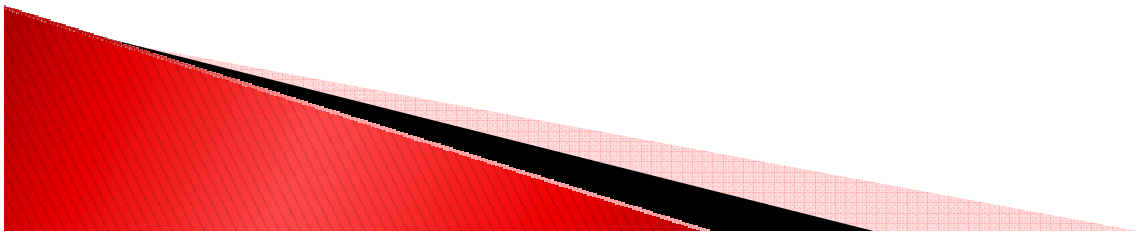
11.6956 Å
11.6844 Å
11.6943 Å

TOTAL	3
MEAN (AVERAGE)	11.69143 Å
STANDARD DEVIATION	0.00613 Å
Variance (Standard Deviation)	0.00004 Å
Population Standard Deviation	0.005 Å
Variance (Population Standard Deviation)	0.00003 Å

This basically explains to you that the c axis is equal to 11.69143 Å ± 0.00613 Å.

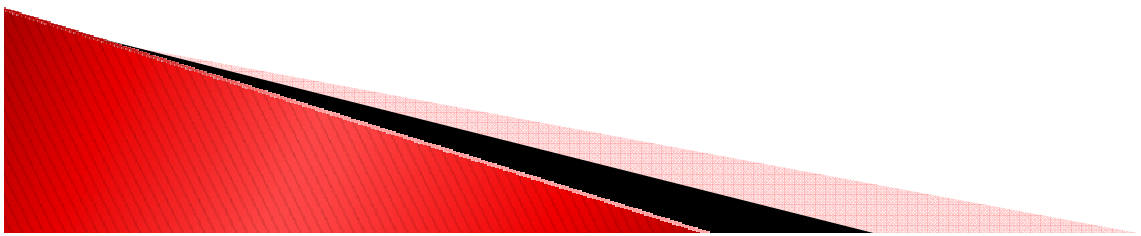
Oxygen Content from C-Axis

δ	0.07	0.27	0.4	0.45	0.52	0.55	0.59	0.62
x	0.93	0.73	0.60	0.55	0.48	0.45	0.41	0.38
a	3.8227	3.8275	3.8349	3.8362	3.8415	3.8433	3.8468	3.851
b	3.8872	3.8875	3.8851	3.8808	3.8778	3.8764	3.8736	3.87
c	11.68	11.706	11.728	11.729	11.747	11.751	11.76	11.771
Tc (K)	90	70	57	57	55	40	15	--



Summary

- Bulk sample of YBCO were synthesized by solid state reaction
- X-Ray diffraction measurements were used to determine the phases present
- No significant impurity levels were observed
- Simulations were used to index all major peaks (C-Program written)
- Oxygen content was determined for c-parameters
- Comparisons with RAMAN and Resistivity show that oxygen content was x~0.9



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