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Superconductivity at IBM - a Centennial Review: Part II – Materials and Physics

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Abstract **- The history of materials and physics research in superconductivity at IBM Research spans a broad range of topics including major contributions in the discovery of organic and high temperature superconductors, elucidation of grain boundary critical currents and magnetic phase diagram, and proposals for the underlying mechanism of high temperature superconductivity.**

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I. INTRODUCTION

The 2011 centennial of the discovery of superconductivity raised global awareness of this exciting and important field. Here we provide a retrospective on the fundamental contributions to this field made by IBM researchers. When viewed over the span of five decades and the three main laboratories of IBM Research, these contributions together form one of the most significant contributions by any one institution. The contributions span new materials discovery, fundamental physics, chemistry and materials science, and technology, and they are highlighted by one of only five Nobel prizes ever awarded in the superconductivity field. This article focuses on materials and physics, while a companion article by Gallagher *et al.*[1] covers the beginning of IBM's research in superconductivity and its sizable applied efforts aimed at superconducting computing and magnetic sensing technologies.

Some of the players in the IBM materials and physics work are shown in Figure 1.

Fig. 1. Some of the players in IBM superconductivity research, at the reception of the Centennial Conference on Superconductivity, Sept. 2011, in den Haag, Holland. Left to right: Jochen Mannhart, first postdoc in IBM Yorktown with Praveen Chaudari and then Research Staff Member in IBM Rüschlikon, now at the Max Planck Institute in Stuttgart, Germany; Alex Müller, IBM Fellow and Nobel Laureate, now at University of Zürich; Alex Malozemoff, Research Staff Member at Yorktown and Research Division Co-ordinator for High Temperature Superconductivity, now consultant to AMSC; Peter Kes, visitor with Chang Tsuei at IBM Yorktown, professor at Leiden and chair of the Centennial Conference; and Alan Kleinsasser, Research Staff Member at Yorktown, now at the Jet Propulsion Laboratory in Pasadena CA.

II. PRE-HTS SUPERCONDUCTVITY AT IBM WATSON LABORATORIES: SOWING THE SEEDS

IBM's earliest basic research in superconductivity took place in the milieu of IBM initial applied effort focused on the cryotron[1]. Arguably the first basic study dates to the end of the 1950's at the IBM Watson Laboratory at Columbia University where Richard Garwin and two Columbia University graduate students, Myriam Sarachik and Erich Erlbach, studied the consequences of the BCS energy gap on the penetration depth [2]. They observed the expected freeze out of quasiparticles at low temperature and inferred a Pb energy gap $2\Delta/k_{\mathrm{B}}T_{\mathrm{C}} = 4.9$, considerably above the BCS (i.e., "weak coupling") prediction of 3.5 and a value of 4.1 inferred from early infrared absorption measurements [3]. (Later a value of 4.6 was determined from careful tunneling studies [4].) Penetration depth studies continued in the early 1960's by IBM theorist Jim Swihart and experimentalist Don Young as the new Yorktown Heights Watson Laboratory was opening in the early 1960's.

Indicative of the prominence of superconductivity within IBM already at this time, an IBM Symposium on Fundamental Research in Superconductivity was held in June of 1961 as part of the dedication ceremonies of the Thomas J. Watson Research Center. Many leading researchers in superconductivity at that time including John Bardeen, Leon Cooper, Bernd Matthias, Ted Geballe, Bill Little, and Michael Tinkham presented at the symposium and contributed written papers to a special issue of the IBM Journal of Research and Development guest edited by IBM theorist Paul Marcus [5]. Figure 2 is a reproduction of an interesting figure shown at the

[1] See Part I [1] for a summary of IBM's research efforts on cryotron devices and circuits.

symposium by Sidney Shapiro and colleagues from Arthur D. Little [6]. What today can be clearly recognized as Josephson current is evident at zero voltage. Bardeen, Cooper, and Phil Anderson were all involved with the meeting, but no one knew what this was at that time. The zero-voltage current was assumed to be some mysterious supercurrent short in the tunnel barrier. Josephson submitted his paper predicting the zero voltage effect on June 8, 1962, a year later [7]. Phil Anderson and John Rowell's paper first claiming observation of the Josephson tunneling effect came about six months after that, with the authors going into some length to explain why the zero-voltage current in their paper was not an artifact [8].

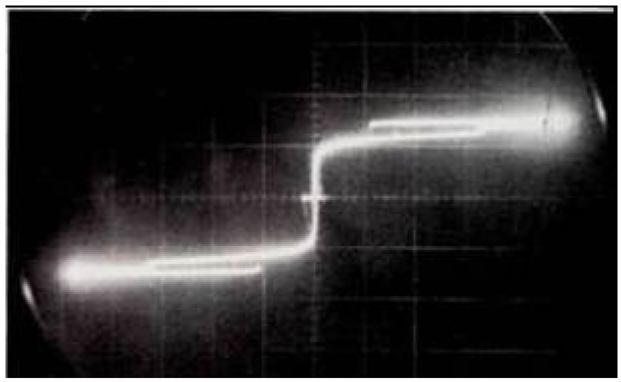

Fig. 2. Photograph of voltage vs. current characteristic of an Al-Al_2O_3-Pb tunnel junction from a paper presented by Sidney Shapiro of Arthur D. Little at June 1961 IBM Symposium on Fundamental Research in Superconductivity. Scale (1 mV/div. vertical; 10 μA/div horizontal). Images taken at 1.28 K and 1.0 K are superimposed and show showing negative conductance at approximately 1.3 mV. A Josephson current can be seen at zero voltage, although it was not recognized as such at the time.

Beginning just after this in the mid 1960's Juri Matisoo began his studies of the switching time of the Josephson effect in what started out as a basic investigation [9] before he turned his attention to the types of circuits that could be made [10]. (See part I of this two part article.) Also around this time Jim Levine was undertaking studies of nonequilibrium superconductors [11], basic studies of what seemed to be potentially be useful effects for IBM applications, although it did not turn out that way.

Beginning in the early 1970's Bob Laibowitz and his group in Yorktown including Chang Tsuei and later John (C. C.) Chi were investigating superconductivity in conjunction with low temperature physics and one of the world's earliest programs developing nanolithography, long before nano became the buzz word that it is today. One of the earliest results by Frank Mayadas and Bob Laibowitz, already in 1972, gave evidence for one-dimensional superconductors [12]. Laibowitz, Tsuei, Eileen Alessandrini and their collaborators deposited some of the highest T_c Nb_3Ge thin films and studied the Josephson effect in Nb_3Ge nanobridges [13, 14], and in work with Dick Voss and Mark Ketchen, they fabricated and studied ultra low noise nanobridge SQUIDS [15,16,17]. Already in 1978, nanostructures with dimensions below 10 nm were fabricated using electron beam lithography with "contamination resist" by Laibowitz in collaboration with Walter Molzen, Alec Broers, Jerry Cuomo, Jim Harper and Erik Harris [18]. On sabbatical in the late 1970's at IBM's Rüschlikon laboratory, which was also working partly on Josephson computer technology, Bob Laibowitz collaborated with Ron Broom and others there to fabricate Josephson junctions with both electrodes being made of niobium [19]. In subsequent work with e-beam lithography, the dimensions of the devices fabricated were reduced to 1 μm^2 and later shown to produce low noise dc SQUIDs [17]. Other collaborations with Rüschlikon fabricated aluminum oxide tunnel junctions on niobium base electrodes, and Praveen Chaudhari's collaborations developed stable Pb alloys.

Collaborations between the IBM Research labs were always encouraged, including sabbaticals, and these often led to very interesting unanticipated outcomes and much more than the simple sum of the parts. One of the most important of seeds for the development of high temperature superconductivity emerged from the two-year sabbatical of Alex Müller at Yorktown in 1979-1981, where he worked primarily with Mel Pomerantz and Bob Laibowitz on microwave properties of superconductors. Müller and Pomerantz, with Alex Malozemoff, then manager of the Magnetism and Superconductivity Department in Physical Sciences, organized an informal study group to discuss Michael Tinkham's book on superconductivity section by section and week by week. During these discussions, Alex Müller revealed his fascination with the remarkable superconductivity in oxides, appearing in materials with very low carrier density, in apparent contradiction to BCS theory. As an expert in the Jahn-Teller effect, which involves large lattice distortion in transition metal oxides, he was also intrigued by the possibility of oxides near the edge of a Jahn-Teller transition providing an unusually large electron-phonon coupling and thus enabling high transition temperatures. These pointers subsequently guided him and Georg Bednorz, back at the IBM laboratory in Rüschlikon, Switzerland, to their amazing discovery. This was one of the rare cases in the history of superconductivity where theoretical ideas led so directly to a major superconductor materials discovery.

Around 1980, Tony Leggett and his collaborators [20] developed interesting ideas on the observation of quantum tunneling in macroscopic systems. The 1 μm^2 all-Nb Josephson junctions were shown in careful dilution refrigerator experiments by Dick Voss and Rich Webb [21, 22] to have their macroscopic junction phase tunnel at the rate predicted by Caldeira and Leggett's macroscopic tunneling theory. These early macroscopic quantum tunneling experiments and later macroscopic quantum coherence experiments provided the basic underpinning for the macroscopic quantum effects that are being vigorously pursued today for quantum computing.

Important materials physics work on amorphous superconductors was also conducted at Yorktown during the early 1980's. The critical current is small but non-zero in these materials – a fact unexpected from a material containing no strong (structural) flux pinning centers. To find out the origin of flux pinning in this class of superconductors, Chang Tsuei, with visitor Peter Kes from Leiden, studied two-dimensional amorphous thin films of Nb_3Ge and found that a large number of weak pins arising from microscopic structural modulations in amorphous superconductors can act collectively to pin a flux line lattice, resulting in a finite critical current [23, 24]. This concept of collective flux pinning was thus experimentally verified more than a decade after it was first proposed by the Russian theorists Larkin and Ovchinnikov.

Other fundamental research included work by Praveen Chaudhari, Bruce Scott and collaborators on thin films of the organic superconductor TTF-TCNQ [25]. John Chi and collaborators developed a novel laser scanning technique to map homogeneity and local dissipation of superconductors [26], a technique which continues to be used widely today to study high temperature superconductors. Near the end of the Josephson computer program there was a realization by the IBM management and technical workers that good science could be done with technology developed by the Josephson computer team. A group in the Physical Sciences Department including John Chi, Art Davidson, Sadeg Faris and Claudia Tesche utilized the high frequency properties of Josephson junctions to develop exploratory high speed electronics, novel switching devices and sensors on a special "Scientific Chip", taking advantage of the Josephson computer technology. A spin-off from this activity occurred during this period as Sadeg Faris left IBM to found Hypres in Elmsford NY. Hypres subsequently manufactured a general purpose Josephson sampling oscilloscope and ultimately became a low-temperature-superconductor device foundry and, though under different leadership, continues today to provide foundry services for developing state-of-the art high-speed superconductor electronics.

After the termination of the Josephson computer program in 1983, a smaller team led by Bill Gallagher was left in place with a two-fold mission: (1) to search for a three-terminal superconducting device that would address fundamental circuit margin problems that always come with two-terminal threshold devices like the Josephson device, and (2) to continue to exploit the scientific potential of Josephson technology in a way similar to the Scientific Chip with a scaled down version [27] of the edge junction technology that the Josephson computer program had developed. Prototype three-terminal superconducting devices were developed [28, 29] including a very interesting FET in which a gate-controlled InGaAs channel was made superconducting by the proximity effect [30], but none of these devices turned out to have the gain and isolation properties needed for digital circuitry. The collective scientific activities produced a number of significant results, including determination of the superconducting energy gap limitations on very fast low-loss signal propagation in superconducting transmission lines [31], demonstrations of low-noise SQUIDs [32] and of multichannel biomagnetic systems incorporating these SQUIDs [33, 34, 35], and demonstration of low noise SIS mixers for radio astronomy [36].

III. PRE-HTS SUPERCONDUCTIVITY RESEARCH IN IBM SAN JOSE

Activity in superconductivity was also vibrant during this period at the IBM San Jose Research Laboratory[2]. In 1970, a small group of scientists, led by George Castro in Physical Sciences, were devoting their efforts to understanding and developing organic photoconductors for use in IBM copy machines (a major IBM product at that time, competing with Xerox copiers based on inorganic photoconductors). As an offshoot of this effort, in 1971 Richard (Rick) Greene started investigating the electronic properties of organic charge transfer salts, based on the molecule TCNQ. Semiconducting TCNQ salts had been discovered at Dupont in the 1960s, but they had attracted little interest in the condensed matter physics community. However, in 1972 a group at Johns Hopkins University synthesized the quasi-one dimensional salt (TTF)(TCNQ) which had metallic properties near room temperature. Shortly after this, Alan Heeger and colleagues at the University of Pennsylvania reported a dramatic increase in electrical conductivity in some crystals of (TTF)(TCNQ) near 60 K, just above the Peierls transition temperature to an insulating state at lower temperatures [37]. This huge conductivity was attributed to superconducting fluctuations and, since the highest known transition temperature T_c in any material at that time was 23 K, this report set off a surge of worldwide interest in organic conductors, not to be duplicated again until the Mueller-Bednorz discovery of high-T_c superconductivity in copper oxides. At IBM San Jose, Greene's solitary effort on organic metals now became the focus of attention for other physicists, such as Paul Grant and Bill Gill, and chemists Bryan Street and Ule Mueller-Westerhoff. Yorktown also became significantly involved with organic metals research,

[2] A separate paper on the San Jose, later Almaden Laboratory may be published in the future (Editor's comment).

led by Phil Seiden, Bruce Scott, Yaffa Tomkiewicz, Ed Engler and Jerry Torrance (the latter two became important contributors to the San Jose effort after 1977).

Experiments at IBM San Jose [38] and elsewhere soon showed that superconductivity was not the cause of the large conductivity at 60 K in (TTF)(TCNQ), and no evidence for superconductivity was found in any organic metal until 1980 when D. Jerôme, K. Bechgaard and collaborators in France discovered superconductivity near 1 K when the quasi-1D organic material $(TMTSF)_2PF_6$ was subjected to high pressure [39]. The French discovery was quickly verified at IBM by Rick Greene and Ed Engler [40] and the era of organic superconductivity truly began. More will be said about this later. Nevertheless, much important and interesting research on the physics of low-dimensional organic metals was carried out at both IBM laboratories and elsewhere (Bell Labs, Penn, Johns Hopkins, Europe and Japan) during this exciting period of 1973-1980 [41].

Shortly after the 1973 Heeger group paper [37], Rick Greene, Bryan Street, and Larry Suter (a Stanford graduate student) discovered superconductivity below 1 K in the inorganic polymer $(SN)_x$ [42,43]. This was a momentous discovery at the time (1975) and it generated significant worldwide interest. Figure 3 shows a picture of Street and Green with a model of $(SN)_x$ from an IBM internal publication just after their discovery. Perhaps the primary reason for this excitement was the theoretical proposal in 1964 by Bill Little [44] that room-temperature superconductivity might be possible by an excitonic pairing mechanism in polymers with attached polarizable molecules. The $(SN)_x$ discovery took the San Jose organic metals group into the additional research area of conducting polymers and Greene became the manager of a large physics and chemistry multidisciplinary effort involving ~15 PhD experimentalists and theorists (including the world renowned John Hubbard). After considerable research at San Jose it was found that $(SN)_x$ was a conventional electron-phonon mediated superconductor and had normal state properties more like a two-dimensional semimetal. Extensive attempts to synthesize compounds analogous to $(SN)_x$ were unsuccessful [45,46]. However, treating $(SN)_x$ with bromine yielded a material $(SNBr_{0.4})_x$ which had an order of magnitude higher conductivity than SN, but the T_c remained essentially unchanged [47,48]. Nevertheless, the IBM San Jose discovery of superconductivity in the $(SN)_x$ polymer started a worldwide research effort to find organic polymers that might be superconducting at higher temperature (none have been found to date).

Fig. 3. G. Bryan Street and Rick Greene showing a model of the superconducting polymer, polysulfurnitride (SNx) in 1975.

A very significant outcome of this organic polymer research was the 1977 discovery of metallic conductivity in the doped polymer polyacetylene, $(CH)_x$, by Heeger, MacDiarmid, and Shirakawa at University of Pennsylvania [49]. This work opened up the whole new field of organic conducting polymers [50] and was recognized for its importance by the award of the Nobel Prize in Chemistry in 2000. Conducting polymers have been developed for many uses, and since 1990 [51], electroluminescent semiconductive polymers have been developed with possible applications as transistors, photodiodes and LEDs. At IBM San Jose, research led by Bryan Street, Art Diaz, Paul Grant and Tom Clarke made important contributions [52,53,54,55, 56] to the understanding and practical development of conducting and semiconducting polymers during the period 1977-1980, including polypyrrole (first introduced by P. Gardini a visiting scholar from Italy) [54]. However, the discovery of the first organic superconductor by Jerôme *et al*.[39] focused research at San Jose back to organic charge transfer salts.

Another significant work on superconductivity at IBM San Jose was the 1982 discovery of superconductivity at 2 K in a second, more two-dimensional, class of organic materials based on the molecule (BEDT-TTF) [57]. This research was a collaboration between Rick Greene, Ed Engler and Stuart Parkin (Rick's postdoc at this time – later to also make major contributions to IBM in developing giant magnetoresistive read heads). This important work showed that organic superconductivity was more widespread than previously believed [58]. The ambient pressure T_c in this class of organic superconductors was eventually raised to near 13 K, far above that found in the quasi-1D organics.

Much novel physics associated with quasi-1D and 2D organic materials was discovered during the 1980-86 period at IBM [59,60]. One important result was the discovery that a small amount of non-magnetic impurity could kill the superconductivity in these organic superconductors [61]. This was the first evidence that the superconductivity in these materials was of an unconventional origin, *i.e*., not simple *s*-wave pairing symmetry or electron-phonon pairing. However, this experiment was ahead of its time, and the issue of non-*s*-wave pairing in superconductors was not considered again until the HTS cuprates were discovered. Now, it is well accepted that cuprates, pnictides, heavy fermions and organics are all unconventional superconductors and electron correlations play a role in the pairing mechanism. Another experimental result of particular note during this 1980-86 time period was the joint IBM San Jose-Sandia Labs discovery [62] of the first example of a magnetic field-induced spin density wave phase transition (FISDW). The lead author of this work was Jim Kwak, a former postdoc at the San Jose lab. This FISDW discovery formed the foundation for many subsequent discoveries of novel magneto-transport physics in organic metals, which continue to this day [63]. Not surprisingly, field induced SDW states have now been shown to play an important role in the physics of 2D systems like the high-T_c cuprates.

IV. DISCOVERY OF HIGH TEMPERATURE SUPERCONDUCTIVITY: THE AVALANCHE

The story of Georg Bednorz and Alex Mueller's 1986 discovery of high temperature superconductivity (HTS) in LaBaCuO [64] (see Figure 4) need not be repeated here. It has been celebrated once again, in 2011, on this 25th anniversary of the original HTS discovery and the 100th anniversary of Onnes' discovery of superconductivity. One of the most astonishing discoveries of contemporary materials science, it has had broad repercussions around the world.

Bednorz and Mueller won the Nobel Prize in 1987, one of the most rapid recognitions in Nobel history. Georg Bednorz was rapidly named an IBM Fellow, joining Alex Muller who had earlier earned this distinction, and the two of them were honored with many other awards.

The discovery electrified the entire scientific world and precipitated an avalanche of research which continues to this day. Hundreds of new cuprate superconductors have been discovered, with ambient-pressure T_c's reaching 135 K. Their properties have been studied exhaustively. Understanding of the phenomenology of superconductivity has been revolutionized. And it seems that every possible fundamental theory of HTS has been proposed, though still with no consensus. Meanwhile applications of HTS are advancing, with major efforts on wires, electric power and magnet applications, and electronic and communications applications. All in all, Bednorz and Mueller's discovery is surely the high point of superconductivity work in IBM and a breakthrough that has rejuvenated the entire superconductivity field.

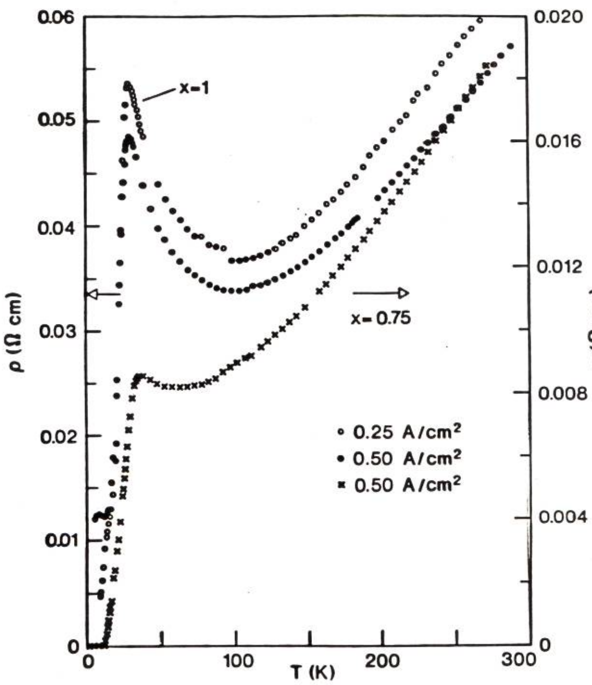

Fig. 4. Historic original resistivity data of Bednorz and Mueller on LaBaCuO in 1986, showing the onset of the superconducting transition above 35 K [64].

As recognition of the significance of Bednorz and Mueller's discovery spread throughout IBM Research in 1986, a huge number of researchers, from Rüschlikon to Yorktown Heights and Almaden, jumped on the HTS bandwagon. This spontaneously generated one of the most significant efforts in the world to understand and develop HTS. Progress occurred at breakneck speed, with exciting results emerging almost daily. As usual during that time period, ATT Bell Labs was a particular focus of competition as Cava and his collaborators already at Christmas time in 1986 announced the discovery of $La_{2-x}Sr_xCuO_4$ with a somewhat higher T_c than the 30 K of Bednorz and Mueller's $La_{2-x}Ba_xCuO_4$. A certain level of competition also raged right within IBM, as researchers raced to find and announce new results, which sometimes appeared in the press even prior to scientific publication. Such were the times!

Worldwide excitement in this field culminated in a jam-packed all night session on March 18, 1987 at the APS March Meeting in New York City, which became known as the "Woodstock of Physics". Results presented focused on the recent discovery of the "YBCO-123" system – the rare earth barium copper oxide with superconductor transition temperature T_c over 90 K. IBM competed with Bell Labs, the University of Houston, and others for the limelight, while Alex Mueller presided over the session in a place of honor. An almost equally raucous occasion, a second Woodstock, occurred at the Interlaken Conference on Superconductivity on Feb. 29 - Mar. 4, 1988, in Switzerland, where researchers gave up outstanding skiing to listen, breathless, as results on the newly discovered bismuth strontium calcium cuprates (BSCCO) and thallium barium calcium cuprates ("Tobacco") were phoned or faxed in to on-stage speakers from their frenetically working collaborators around the globe. These last minute messages included the latest results on the thallium-based superconductors transmitted to IBM speaker Paul Grant from Ed Engler and his IBM colleagues at Almaden. Researchers' temperatures and new critical temperature records rose in unison during that unforgettable week!

To bring some perspective to this creative chaos in IBM, Praveen Chaudhari, then director of Physical Sciences at Yorktown, appointed Alex Malozemoff as Research Division Co-ordinator for High Temperature Superconductivity, who in turn worked with Paul Grant leading the Almaden effort, and with Alex Mueller, Georg Bednorz and their colleagues in Rüschlikon. Malozemoff organized regular meetings at Yorktown, with frequent visitors from the other sites, to share results and develop collaborations. He assured management support for the broad-ranging activity, and traveled around the various IBM divisions, updating them on the progress and defining the potential implications for their business areas.

In mid 1987, Malozemoff led an IBM task-force on potential HTS applications, with results summarized in an article with Bill Gallagher and Bob Schwall [65]. For power cable applications, this article built on the early path-breaking study by IBM's Dick Garwin and Juri Matisoo on the possibility of gigawatt-level 4.2 K DC power cables [66]. At that early time when HTS wires and Josephson junctions were not even demonstrated yet and 77 K current densities were still very low, the task force presciently saw the real opportunity for HTS magnets and HTS ac power cables. Indeed, this opportunity ultimately drew Malozemoff and Schwall away from IBM to join American Superconductor (now AMSC), where they spearheaded the development of HTS wires and magnets. This is one of the major practical outflows from Bednorz and Mueller's discovery: today, robust and flexible, high current HTS wires are commercially available, and HTS ac power cables have been demonstrated around the world in use in the power grid.

The task force also recognized the enormous challenges facing application of HTS in data processing, either in a next generation Josephson computer or in ultra-low-loss connections in chip packaging. Neither of these has, in fact, materialized, and eventually this led, after many years, to IBM's loss of interest in superconductivity and the gradual dissolution of the superconductivity effort, beginning in the 1990's, although work continues on superconductor-based quantum computing [1]. Many of IBM's former technical leaders in the field now populate universities, national labs and industries where significant impact of the IBM work continues. Nevertheless, the achievements at IBM during that exciting first decade after the discovery of HTS stand as a great contribution to science.

V. HTS BULK SYNTHESIS AND STRUCTURE IDENTIFICATION

The first major technical breakthrough of the IBM activity, after Bednorz and Mueller, came in Almaden, where the team of Paul Grant, Robbie Beyers, Ed Engler, Stu Parkin and their collaborators worked around-the-clock to identify, separate and characterize the superconducting phase in the mystery compound of yttrium, barium, copper and oxygen reported in early 1987 by Wu and Chu et al. of the Universities of Alabama and Houston [67]. Using electron microprobe and energy dispersive x-ray analysis on the solid-state-synthesized multiphase mixtures reported by Wu and Chu's teams, they identified the green "211" phase and the all-important superconducting black "123" phase [68]. They followed by synthesizing the pure compound and identifying the remarkable 123 structure with its copper oxide chains and planes. A beautiful study of the lattice parameters as a function of temperature, carried out by a collaborating team from Almaden and Yorktown, revealed the remarkable tetragonal-to-orthorhombic transition at 670 C, key to understanding the essential role of oxygen during processing in achieving the fully superconducting $YBa_2Cu_3O_7$ (YBCO-123) [69]. This activity raced day by day with the equally determined effort at Bell Labs.

The battle for patent leadership is an interesting sidelight of the superconductivity story at IBM. The Almaden group filed its patent on YBCO-123 in March 1987, a mere three days after the ATT Bell Labs group, but with the correct oxygen processing specification. Along with patent applications from the University of Houston and the Naval Research Laboratory, these filings led to one of the most contentious and protracted interference battles in US Patent Office history. The final result was a disappointment for IBM – over a decade later, the patent on YBCO-123 issued to Lucent, then owner of Bell Labs. But finally in 2011, IBM was granted in the United States the fundamental patent by Bednorz and Müller on oxide superconductors containing a rare earth or yttrium, an alkaline earth and copper, which ended up covering as an umbrella not only the original LaBaCuO material but also YBCO [70].

Creative synthesis juices started flowing again at Almaden in early 1988 when Maeda in Japan [71] announced the novel HTS system based on bismuth, strontium, calcium cuprate, and when Sheng and Hermann at the University of Arkansas [72] announced the thallium-cuprate superconductors. Ed Engler with Stuart Parkin and collaborators at IBM Almaden quickly synthesized new members of these materials, achieving, albeit fleetingly, a new world record T_c of 125 K in a $Tl_2Ca_2Ba_2Cu_3Ox$ thallium cuprate composition [73]. The team that produced the initial 125 K superconducting composition is pictured in Figure 5.

In the mid '90s, by using the techniques of atomic-scale mixing and precise annealing in mercury and oxygen atmospheres, Chang Tsuei and his co-workers overcame various experimental difficulties and succeeded in fabricating the first Hg-based cuprate ($HgBa_2CaCu_2O_{6+\delta}$) films that exhibit zero-resistance at 125 K [74,75,76]. These Hg-based films have extremely high current carrying capability at high temperature (for example, zero-field J_c = 10^6 A/cm^2 at 100K). This is very close to the ultimate theoretical limit set by BCS depairing. SQUIDs based on these films, developed by Lia Krusin-Elbaum and collaborators at Yorktown have been demonstrated to operate up to a record-setting 112 K [77].

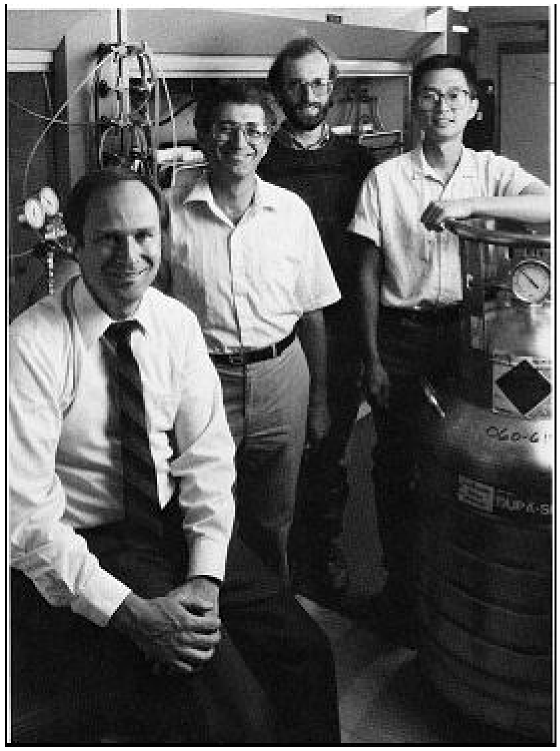

Fig. 5. Ed Engler, Adel Nazzal, Stuart Parkin, and Victor Lee in 1988 when they produced the then world-record highest temperature superconductor $Tl_2Ca_2Ba_2Cu_3Ox$ with a T_c of 125 K.

VI. HTS SINGLE CRYSTALS AND FILMS

At Yorktown, Tim Dinger and then Fred Holtzberg led efforts which produced the world's most perfect single crystals of YBCO-123. Dinger's initial production of 0.5 mm single crystals was the serendipitous but reproducible outcome of the way he and collaborators prepared some of the first 90 K YBaCuO ceramic samples in Yorktown. Those samples, prepared with Bill Gallagher and others, had grains big enough to allow TEM lattice imaging of the structure by Tom Shaw others [78]. The first single-crystal magnetic characterization by Dinger with Tom Worthington, Bob Sandstrom, and Bill Gallagher showed a large supercurrent density anisotropy between the ab-planes and c-axis [79]. Subsequent careful characterization Worthington,, Gallagher, and Dinger gave the first set of anisotropic parameters for the YBaCuO material [80], and started to clear up a lot of the confusion surrounding all earlier high-T_c measurements, which had been made on polycrystalline materials. Fred Holtzberg working with post doc Debbie Kaiser soon improved on the growth of single crystals [81] and for a short time IBM found itself with a bit of scientific monopoly on single crystal $YBa_2Cu_3O_{7-x}$ samples. This enabled a series of highly cited papers on the first characterization of the anisotropic resistivity [82], anisotropic energy gap inferred from infrared energy absorption data [83, 84, 85], and the first anisotropic tunneling data [86, 87].

Fig. 6. Bob Laibowitz and the late Roger Koch shown in 1987 taking resistive transition data for an epitaxial YBaCuO superconducting film.

Another development of major significance, for both physics and future applications, was the synthesis of the first epitaxial YBCO thin films by Bob Laibowitz, Roger Koch, Praveen Chaudhari and Dick Gambino using e-beam evaporation on polished strontium titanate single-crystal substrates [88]. They demonstrated for the first time high current densities of order 1 MA/cm^2 at 77 K, a major step in enabling future practical applications. The thin film architecture also enabled many important new studies of the YBCO and other HTS materials. Figure 6 shows Laibowitz and Koch taking a resistance temperature curve for a high-T_c film sample.

One of the most important outflows of these thin film breakthroughs was the brilliant work of a team led by Praveen Chaudhari and including postdoctoral visitors Duane Dimos and Jochen Mannhart [89, 90, 91, 92] in elucidating the mechanism and systematics of current flow across grain boundaries in HTS materials. Building on an earlier suggestion at the Woodstock meeting by Mas Suenaga of Brookhaven National Lab, Alex Mueller and Guy Deutscher of Tel Aviv University had already published a fundamentally important Physical Review Letter [93] highlighting the impact of the short HTS coherence length on grain boundaries, making them weak links and hence major obstacles to current flow. And it was already known that polycrystalline materials, with their omnipresent grain boundaries, conducted supercurrent at very low current density.

Dimos, Chaudhari and Mannhart conceived of a clever way to study this problem: they first sintered together two strontium titanate crystals to form a bicrystal with a known grain boundary angle; then they polished the surface and grew YBCO on this surface epitaxially, replicating the grain boundary. With their colleagues, they then made the first direct measurement [89] of the critical current densities J_c in single YBCO grain boundaries, which were as much as two orders of magnitude smaller than the intragrain J_c adjacent to the grain boundary measured. This finding confirmed the origin of the great disparity in J_c between polycrystalline and single-crystal cuprate superconductors. Furthermore, this work also revealed the Josephson weak-link nature of the grain boundary junctions, enabling demonstration of the first dc SQUID prototype made of YBCO grain boundary weak links and leading to some basic patents. This method of fabricating HTS junctions continues to be used widely today.

A laborious set of experiments on a series of controlled grain boundary angles then revealed another astonishing result: the exponentially decreasing dependence of J_c on increasing grain boundary angle (see Figure 7) [91]. In addition to its fascinating scientific ramifications [92], this work demonstrated the need to keep grain boundary misorientations below about 4 degrees to achieve single-crystal-like J_c and hence the need for highly textured YBCO if one wanted a long high-J_c polycrystalline wire. Ultimately this work gave birth outside of IBM to a novel type of superconductor wire, deposited on tape-shaped substrates with a highly textured surface on which the YBCO superconductor is grown epitaxially [94]. Astonishingly high J_c (>3 MA/cm^2 at 77 K) over hundreds of meters have been achieved in this robust and flexible tape-shaped wire, called second generation or coated conductor wire, making it the leading type of wire today for magnet and electric power grid applications; it is now sold commercially by a number of companies.

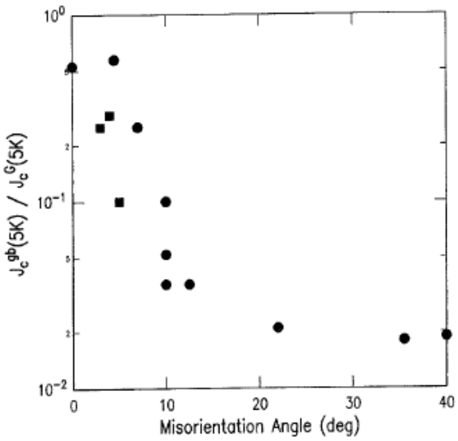

Fig. 7. Historic results of Dimos, Chaudhari, and Mannhart in 1990 [91] on YBCO films grown on bicrystal substrates, showing the dependence of YBCO grain boundary current density, normalized by the intragrain current density, as a function of grain boundary misorientation angle at 5 K. These results stimulated the subsequent development of the HTS coated conductor technology using highly textured YBCO thin films grown on textured templates.

In addition to the ground-breaking development of flux-grown single crystals and e-beam deposition of HTS films, many other novel synthetic processes were pioneered at IBM Yorktown. The groups of Jerry Cuomo and Bob Laibowitz were among the first to develop magnetron sputtering and pulsed laser deposition of HTS films [95,96]; both processes have since been widely used for research and practical application. And in 1988, Arunava Gupta and his collaborators reported the successful YBCO film formation in an *ex-situ* process by first coating metal trifluoroacetate precursors and subsequently annealing to first drive off the carbon and then to form the superconducting phase [97]. This process has since formed the basis of one of the successful YBCO coated conductor wires produced by AMSC [94]. Another important process developed by the Cuomo group should be mentioned, though it was initially not carried out on HTS materials: this is what has become known as ion beam assisted deposition or IBAD [98]. Subsequently it also became used to texture buffers for the YBCO coated conductors and is widely used by companies like Fujikura and SuperPower [94].

VII. HTS MAGNETIC PROPERTIES AND PHASE DIAGRAM

IBM researchers at Yorktown initiated a revolution in understanding HTS magnetic properties and the phenomenology of superconductivity. Mueller and Bednorz already recognized that something unusual was afoot in the magnetic properties of the cuprates, and they ventured a first explanation in terms of the glassy behavior of a random weakly-linked collection of superconductor grains [99]. However, in a ground-breaking paper [100], Alex Malozemoff, working with postdoctoral visitor Yosi Yeshurun from Israel's Bar-Ilan University, reported logarithmic relaxation of magnetization and current in an YBCO *crystal*, which eliminated any explanation based on polycrystalline grain boundaries [99]. They recognized that this phenomenon could be explained in terms of flux creep. While flux creep had been known in low-temperature superconductors, it was a tiny effect. In HTS the effect was on a giant scale, because of the much greater thermal activation at higher temperature, hence the term "giant flux creep."

This insight opened up a new explanation of HTS magnetic properties: using the conventional Bean critical state model, but with a time-relaxed critical current density as flux lines are thermally activated out of their pinning wells. Tom Worthington and co-workers' [80] ac experiments on the YBCO crystals had identified a line of susceptibility anomalies in the *H-T* plane, which was anisotropic for fields along the different principal directions. Worthington also found that the temperature of the susceptibility anomaly depended logarithmically on frequency. Originally thought to be a measure of the upper critical field H_{c2}, the susceptibility anomaly was reinterpreted by Malozemoff, Worthington, Yeshurun, Holtzberg and visitor Peter Kes from Leiden as an irreversibility line whose frequency dependence could be accounted for by the thermal activation model [100]. They recognized that the actual upper critical field H_{c2} lay at fields far above this irreversibility field at any given temperature, so that a large region in the *H-T* plane between H_{c2} and the irreversibility line constituted a novel phase of unpinned, liquid vortices (see Figure 8) [101]. This new phase diagram created a revolution in the understanding of superconductor phenomenology and spurred worldwide activity in the novel field of "vortex matter." For his contributions to understanding vortex physics and applying this knowledge to the subsequent development of HTS wires, Malozemoff was awarded the IEEE Applied Superconductivity Award in 2011.

This new understanding of the magnetic phase diagram by the Yorktown team was quickly followed by their determination of the lower and upper critical fields [101] and a determination of the temperature-dependent magnetic penetration depth by Lia Krusin-Elbaum, Rick Greene and their collaborators [102]. Key theoretical work was contributed by Matt Fisher to develop a powerful and fundamentally new concept for the nature of the superconductor phase transition in the context of thermal activation and random pinning of flux lines: vortex glass superconductivity [103]. This work enabled a successful scaling of the transport properties studied by Roger Koch, working with Bill Gallagher, Arunava Gupta and others [104]; this approach has become standard worldwide in analyzing transport data on HTS materials. . Other visitors at IBM, including Eli Zeldov and Nai-Chang Yeh also contributed to the understanding of the transport properties.

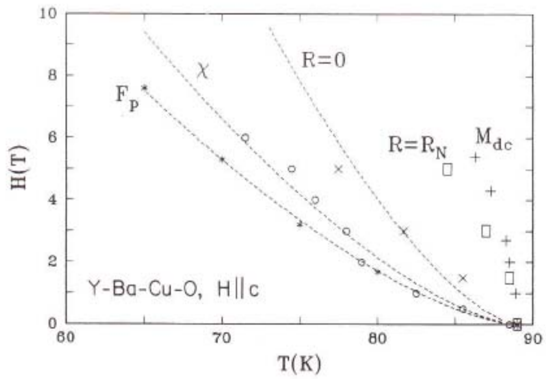

Fig. 8. Magnetic susceptibility χ anomalies [80], pinning force F_p limits and zero-resistivity in YBCO crystals in the field-temperature plane, along with onsets of DC diamagnetism and deviation from the normal resistance R_N. Malozemoff *et al.* [100] interpreted the first three as irreversibility lines and the last two as approximate measurements of the upper critical field, opening up a new understanding of the HTS phase diagram and launching a revolution in the field of vortex matter.

Fisher's theory provided a novel interpretation of the index value *n* in the *V-I* curve ($V \sim I^n$), which Malozemoff and Fisher found to be remarkably universal, around 30 for YBCO-123 in a variety of forms – bulk, films, crystals, etc [105]. They also found a temperature independent normalized relaxation rate *dlnM/dlnt* of about 1/30, suspiciously the inverse of the index value. In earlier work on low temperature superconductors, a power law dependence of *V* on *I* had always been interpreted as an indication of material non-uniformity. The vortex glass theory now offered a radically new interpretation, at least for highly uniform HTS materials: it was another consequence of flux creep in a glassy limit. The theory predicted the normalized relaxation rate to be just *1/n* in agreement with experiment, and determined it to be given by a glassy exponent times the logarithm of the flux line hopping frequency [105].

The recognition of the importance of pinning in determining critical currents led to the first irradiation work on YBCO crystals: Visitor Leonardo Civale, with Alan Marwick, and others irradiated YBCO crystals with heavy ions to create columnar defects, which acted as strong pinning centers for the linear flux lines [106]. Collaborating with Jim Thompson of the University of Tennessee, Lia Krusin-Elbaum and her colleagues also discovered the surprising phenomenon that the parallel damage columns enhance flux creep in an intermediate temperature range [107]. Krusin-Elbaum and her collaborators at Yorktown then took this work to the next level, finding that splayed columnar defects are much more effective than parallel columns because they suppress free motion of flux line kinks straddling two columnar defects [108].

A ground-breaking study led by Jerry Torrance and his collaborators in Almaden, charted for the first time the superconductor phase diagram as a function of hole concentration *p* in $La_{2-x}Sr_xCuO_4$, showing a characteristic parabolic curve, first rising, then falling as a function of *p*, with the insulating magnetic phase on the left-hand side and a normal metal on the right-hand, high-*p* side [109]. A follow-up study with his postdoc Tokura, now a distinguished scientist in Japan, established the same kind of chart for $YBa_2Cu_3O_{7-x}$ [110].

VIII. HTS PAIRING SYMMETRY AND MICROSCOPIC MECHANISM

Other work at Yorktown focused on a fundamental understanding of the microscopic nature of high temperature superconductivity. Dennis Newns, Chang Tsuei and their collaborators employed a Van Hove singularity model to understand the essential properties of cuprate superconductors [111, 112, 113, 114, 115, 116, 117]. A Van Hove singularity (VHs) manifests itself as a peak in the electronic density of states (DOS), coinciding with a saddle-like region in the energy surface on which an electron moves. As a consequence of topology, there is at least one VHs present in a two-dimensional band structure. The Van Hove model developed at IBM considered the close proximity of the Fermi level to a VHs and predicted that, within the BCS formalism, the energy-dependent DOS near a VHs can give rise to an enhanced T_c, reduced isotope effect, *etc*., as observed in cuprate superconductors with the T_c – optimum compositions. Furthermore, when the Van Hove condition is fulfilled, the large phase space available for electron-electron scattering at a VHs leads to marginal Fermi liquid behavior – the quasiparticle lifetime scales linearly with quasiparticle energy as observed in photoemission and infrared conductivity.

More recently, high-resolution ARPES measurements have confirmed the existence of an extended VHs in the energy surface close to the Fermi level. Furthermore, theoretical studies, based on various 2D Hubbard models, have shown that such flat band features in the band structure can be attributed to the strong correlation in cuprates. The Van Hove work at IBM also shows that, as the Fermi energy is shifted by doping away from VHs, the anomalous properties such as high-T_c, small isotope effect and marginal Fermi liquid behavior should revert to the conventional Fermi liquid properties, in good agreement with experiment.

Twenty-five years of extensive research have yet to produce a general consensus on the origin of high-temperature superconductivity. Recently, mounting evidence, *e.g*., from the in-plane isotope shift studies and other experiments has led to a re-examination of the role of phonons. In a novel approach, Newns and Tsuei [118] find that the interaction responsible for pairing in cuprates depends on the stability of the Cu-O-Cu bond in the CuO_2 plane. The new model is based on the observation that the charge carrier motion along the in-plane Cu-O-Cu bond must be non-linearly modulated by the oxygen vibrational degrees of freedom, enabling *d*-wave pairing mediated by an anharmonic two-(local) phonon process. This fluctuating bond pairing mechanism, decoupled from the large on-site Coulomb repulsion, is qualitatively different from the conventional one-phonon BCS pairing interaction. As shown in a recent paper, this model naturally explains several salient HTS features including intrinsic *d*-wave pairing symmetry, doping-dependent T_c, isotope effect, pseudo-gap state, and the recently observed nanoscale inhomogeneity and C4 symmetry [119].

An unambiguous determination of the pairing symmetry in cuprate superconductors is critical for understanding the origin of high-temperature superconductivity. With this goal, Chang Tsuei and collaborators designed a clever phase-sensitive tri-crystal experiment, using a c-axis epitaxial film consisting of three cuprate crystals with specially-chosen crystallographic directions [120]. The three linear interfaces between the three crystals form three grain boundary Josephson junctions. The tricrystal geometry was chosen by design so that for a $d_{x^2-y^2}$-wave superconductor, there would be an odd number of sign changes in the component of the pair wavefunction normal to the grain boundaries, and hence a net phase shift of π in a superconducting loop circling the tricrystal meeting point. The magnetic flux threading through such a loop (termed a π-loop) is thus quantized in *half-integral* multiples of the flux quantum Φ_o.

Tsuei and his collaborators who developed the tricrystal sample, working with John Kirtley and Mark Ketchen, who had developed a high-resolution scanning SQUID microscope, made the first *direct* observation of the half-flux quantum effect in $YBa_2Cu_3O_7$ (YBCO) in 1994 [120], a definitive signature of *d*-wave pairing symmetry (see Figures 9 and 10).

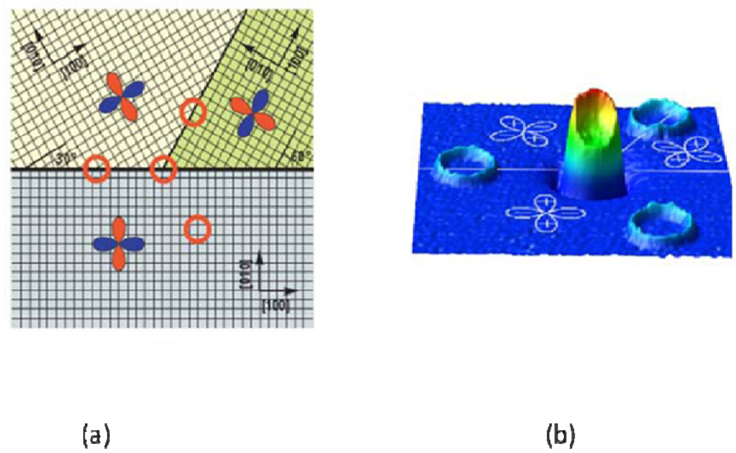

Fig. 9. a) Schematic of tricrystal geometry for *d*-wave pairing symmetry test, with patterned cuprate rings shown schematically as circles and in-plane *d*-wave lobe orientations indicated in each grain. b) SQUID scanning microscopy (SSM) reveals a spontaneously generated half-integer flux quantum in the ring at the tricrystal intersection and, as the experimental control, zero flux in all other rings. This observation was the first definitive evidence for *d*-wave pairing symmetry of high-temperature superconductors [119,122].

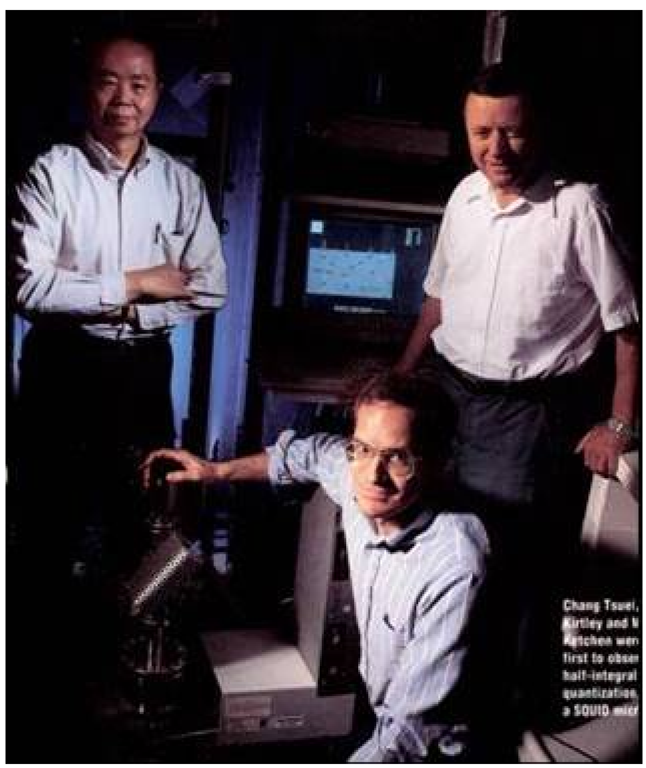

Fig. 10. Chang Tsuei (left) shown with John Kirtley (center) and Mark Ketchen at the scanning SQUID microscope.

They followed this historic result with series of phase-sensitive tests using tricrystal experiments to demonstrate $d_{x^2-y^2}$ pairing symmetry in three different cuprate systems with doping levels ranging from underdoped, through optimal doping, to the overdoped regimes [121, 122]. The results of this work clearly show that the $d_{x^2-y^2}$ pair state is robust against a wide-range of doping variations. Furthermore, they demonstrated that the *d*-wave pair state is robust against time-reversal symmetry breaking and a large variation in temperature up to T_c. Together with the SQUID-based experiments by Van Harlingen and collaborators at the University of Illinois, the IBM work has convincingly settled the decade-long *s*-wave vs. *d*-wave debate in favor of exclusive $d_{x^2-y^2}$ pairing symmetry in all the cuprate systems investigated [123, 124].

The work on *d*-wave pairing symmetry has also inspired novel applications of *d*-wave superconductors, including a proposal to do quantum computing with *d*-wave superconductors [125]. More recently, Tsuei *et al.* have fabricated all-*d*-wave π-SQUIDs [126, 127] and periodic arrays of 2×10^4 half-fluxons integrated on a single chip. This work opens the door to more fundamental studies and potential applications utilizing the $d_{x^2-y^2}$ gap symmetry.

The standard phase-sensitive pairing symmetry test, developed for the cuprate superconductors, does not apply readily to recently discovered pnictide superconductors based on Fe and As. The heart of the problem lies in the fundamental difficulty associated with their complex multi-orbital physics and Fermi surface structure, the lack of high-quality epitaxial films or of large single crystals, and the difficulty in fabricating Josephson junctions. Ching-Tzu Chen, Tsuei, and Ketchen reported a novel *phase-sensitive* method for probing unconventional pairing symmetry in the *polycrystalline* iron-pnictides [128]. They managed to circumvent the aforementioned problems by investigating the quantized flux states in a superconducting niobium/iron-pnictide loop. Through the observation of integer and *half*-integer flux-quantum transitions, they found evidence for *s*-wave pairing with a *sign change* in the superconducting order parameter in the one-layer $NdFeAsO_{0.88}F_{0.12}$ superconductor.

For their seminal work on the HTS pairing symmetry, Tsuei and Kirtley won the 1998 Buckley Condensed Matter Prize of the American Physical Society.

IX. CONCLUSION

IBM Research's superconductivity work has spanned more than fifty years, exploring the cryotron and Josephson computing, pioneering SQUIDs for ultrasensitive measurement, exploring macroscopic quantum interference and coherence for quantum computer applications, and vitalizing global superconductivity research with the discovery of HTS as well as building the foundation for subsequent applications in electronics, magnets and electric power, and perhaps quantum computing. Today IBM continues with significant activity aimed at developing coupled superconducting qubits for quantum computing. These major contributions continue to impact global superconductor research and application.

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The next pages present the short biographies of co-authors.

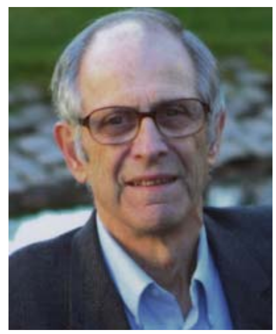
About the Authors:

Dr. William J. Gallagher's biography is included in the Part I of this paper.

Dr. Richard L. Greene was born August 26, 1938 in Bridgeport, CT. He received his B.S. in Physics from M.I.T. (1960), was Engineering Officer in U.S. Navy, 1960-62 and graduated with Ph.D. in Physics at Stanford University (1967). He was Research Associate, Stanford University, 1967-70; Research Staff Member and Manager, IBM Research Division, San Jose, CA and Yorktown Heights, N.Y., 1970-89; Visiting Professor, University of Grenoble, France, 1978-79; Director, Center for Superconductivity Research, University of Maryland, 1989-2007. He is currently Professor of Physics at University of Maryland, College Park. Dr. Greene is Fellow of the American Physical Society and of the American Association for the Advancement of Science (AAAS). He served as Chairman of APS New Materials Prize Committee, 1986; on the Executive Committee of the APS Division of Condensed Matter Physics, 1992-95 and on APS Apker Award Committee, 2000-04. In 2012 he has been Chairman of the Program Committee for the tri-annual Materials and Mechanisms of Superconductivity Conference (M2S-2012); Honored by Thomson-ISI as a Highly-cited Physicist, 1981-2001, with current h-index 70. He has co-authored over 350 publications.

Richard L. (Rick) Greene, 2009

Dr. Robert B. Laibowitz received Bachelor degrees in both Arts and EE and Masters degree (EE) from Columbia University and his Ph.D. in Applied Physics from Cornell University in 1967. He has worked at the IBM Research Division for many years specializing in device development and material characterization in such fields as tunnel junctions, MOS capacitors, ferroelectrics, phase change , superconductivity and a variety of dielectric measurements (including both high and low k). He worked on the Josephson Computer project during 1974-1976 at the IBM Zurich Laboratory. He joined the EE Dept at Columbia University in 2002 as a Senior Research Scientist and Adjunct Professor. His current work concerns leakage (conduction) mechanisms, trapping, barrier heights and interfaces, reliability and time dependent dielectric breakdown in low k dielectrics. He has also consulted on materials aspects of quantum computing at IBM. He has authored or co-authored about 175 peer reviewed papers and 35 patents. He is a Senior Member of the IEEE and a Fellow of the American Physical Society.



Robert B. Laibowitz, 2009

Dr. Alexis P. Malozemoff received a B. A. from Harvard University in Physics and Chemistry, and a PhD from Stanford University in Materials Science. For 19 years, he worked at IBM Research as staff scientist, manager and senior manager in magnetism, superconductivity and condensed matter physics. He co-discovered "giant flux creep" and the irreversibility line in high temperature superconductors (HTS), key factors determining the maximum current a superconductor can carry. He is also known for his work on magnetic materials, including his theory of exchange anisotropy, and his discoveries related to magnetic bubble and recording technologies. Dr. Malozemoff joined American Superconductor (AMSC) in 1991, serving as Chief Technical Officer till his retirement in 2009, managing HTS wire and applications development, external collaborative programs and patent strategy. Fellow of the American Physical Society and IEEE, he has served on various conferences, national committees and advisory boards, and co-chaired the U. S. Department of Energy BESAC Subcommittee on Science for Energy Technology, completing its report in 2010 (http://science.energy.gov/~/media/bes/pdf/reports/files/setf_rpt.pdf). In 2011 he won the IEEE Award for Continuing and Significant Contributions in the Field of Superconductivity. He has authored over 200 publications, multiple patents, book chapters and review articles, and a book *Magnetic Domain Walls in Bubble Materials* with J. C. Slonczewski,

Alexis P. Malozemoff, 2011

Dr. Chang C. Tsuei received his B.S. in 1960 from National Taiwan University, M.S. and Ph.D. from California Institute of Technology in 1963 and 1966 respectively. He joined IBM in the Physical Sciences Department of Thomas J. Watson Research Center as Research Staff Member in 1973. During 1974 – 1993, he held several research manager positions responsible for research programs in superconductivity, physics of amorphous materials, and physics of structured materials. Since 1993, he has returned to full time research to study the fundamental aspects of high-temperature superconductivity in cuprates. He is currently interested in the topological nature of d-wave superconductor-based π-loops. He is Honorary Chair Professor in Physics of National Tsing Hua University since 2006. He has been a Fellow of the American Physical Society since 1974 and a Fellow of the American Association for the Advancement of Science since 2001. He was elected academician of Academia Sinica in 1996. He received a Max Planck Research Prize from Max Planck Society of Germany in 1992. For his work on elucidating the pairing symmetry of high-temperature superconductors, he was awarded the 1998 Oliver E. Buckley Condensed Matter Physics Prize of the American Physical Society, and an IBM Corporate Award in 2000.

Chang C. Tsuei, 2007