

A Random Walk Toward High-Speed Sample Spinning

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My introduction to ESR and NMR began under Charlie Poole¹ in the graduate Physics Department at the University of South Carolina. There I became rather well known throughout both the Physics and Chemistry Departments as being unafraid to walk into any instrumentation laboratory and start dismantling equipment. This brazen behavior was tolerated because I would put the instruments back together with improved gadgets I had made (usually without permission) in the machine shop and electronics laboratories.

One spring day in 1979, Paul Ellis (*Multinuclear MR Experiments, the Surrogate Probe Strategy, and Other Fun & Games*) came looking for me. He needed some kilowatt UHF transmitters and a high-speed sample spinner that really worked to perform his solid state NMR experiments. At the time I was more confident with the transmitters, being well versed in the witchcraft of microwave and rf from ESR and years of TV repair experience. But I had also been rebuilding automobile and tractor engines since early childhood and was keenly interested in turbomachinery—with visions of the Brayton cycle achieving 65% energy conversion efficiency dancing in my brain.

After completing the transmitters and struggling for a few months with the Andrew-Beams spinners, I came to the conclusion that the problems with low-frequency torsional modes (several hundred Hz) and frequent instabilities above 2 kHz could only be solved by going in the general direction that Lippmaa, Veeman, Pines, and others were going: double cylindrical air bearings (see *Solid State Probe Design*). Paul wanted to look at natural abundance ¹³C in surface-adsorbed species, so it was necessary to explore the use of high-strength ceramic rotors for improved filling factor (see *Probe Design & Construction*). We found operation at moderately high spinning speeds to be much more difficult than the early papers seemed to imply—at least with the higher density ceramic rotors. Catastrophic explosions from materials defects were common, so I became very experienced in the precision diamond grinding of hard ceramics. On more than one occasion I would work through the night in the machine shop (my wife Judy would sometimes relieve me at the lathe) so that the NMR experiments could continue the next day.

However, we could not blame all the crashes on ceramics defects, so it was necessary to do a more careful study of gas bearings and microturbines. It was encouraging to find calculated optimum bearing hole size and radial clearance reasonably close to experimentally determined optima, but that didn't make it any easier to eliminate systematic and statistical measurement errors or to reproduce the required dimensions.

Simple calculations eventually convinced us that we were often limited by the 'whirl instabilities' previously reported in gas bearings for dental drills. In church choir one Sunday, while half-doing and half-listening to the resonant bass from

the organ, I worked out the increase in the conical whirl mode that could be expected simply by changing the turbine caps from Lippmaa's design (internally hollowed and filled with sample) to externally hollowed and empty.² The improvement proved to be greater than I had expected because of the additional beneficial effect on damping. After another year of constructive criticism from Ruth Inners, Paul Ellis, and J. B. Spitzmesser, my CP MAS probes appeared to be ready for a small commercial venture. In the spring of 1982, a physics colleague Bruce Sofge succeeded in convincing a wealthy friend to loan us approximately \$100 000. Judy, J. B., Bruce, and I started Doty Scientific, Inc. So it began.

In June 1986 at the EENC in Bad Aussee, Vagn Langer and Preben Daugaard upset the status quo by demonstrating the design they had developed for Varian with reproducible spinning at surface speeds above 70% of the speed of sound.³ Vagn was concerned that Varian would now put me out of business. I assured him that he needn't worry. The key difference was obvious upon casual inspection: the gas from Vagn's air bearings was allowed to exit axially in both directions, nearly doubling the area of the effective hydrostatic bearing and hence the radial stiffness. Indeed, this had been done by others before (so no problem with patents), but I wasn't doing it. A few months later, our high speed model emerged, and we were spinning routinely at 75% of the speed of sound. Vagn was relieved—Varian was dismayed.

My earlier work in energy conversion had introduced me to one of the great turbine engineers, David Gordon Wilson.⁴ In the spring of 1990, I called him up and asked him if he would be interested in collaborating on the development of an efficient microturbine. The ensuing relationship was one of the more rewarding of my business career. Most of the final supersonic spinner design details were worked out and communicated back to the plant by fax during the EENC in Veldhoven that May, and within weeks we had a manufacturable design approaching 90% of the speed of sound. A few months later we were achieving supersonic surface speeds and isentropic turbine efficiencies above 30% for 7-mm rotors (compared with 8% for our previous 7-mm microturbines), but scaling to smaller sizes proved to be nontrivial. The following summer while discussing with David the difficult resonances in our 5-mm spinner, we found time for a pleasant evening of singing old English madrigals with his wife around the grand piano in his living room in Cambridge. It was to take two more years and hundreds of failed experiments by my patient colleague Jerry Hacker during difficult recessionary times before we were to recognize several nonlinear scaling laws and the importance of the Bernoulli effects over the ends of the turbine blades and achieve routine spinning above 24 kHz with 3.5-mm rotors.

REFERENCES

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Biographical Sketch

F. David Doty. *b* 1950. B.A., 1972, Physics, Anderson University, Indiana, USA, Ph.D., 1982, Physics, University of South Carolina. Began work in NMR under Paul Ellis. President of Doty Scientific, Inc., 1982 to present. Approx. 20 publications and 15 patents. Research interests include rf electronics, NMR probe technology, electromagnetism, manufacturing turbomachinery, energy conversion cycles, ceramics engineering.