Historical Records of Australian Science, 2015, 26, 179–191 http://dx.doi.org/10.1071/HR15006

# Bruce Godfrey Hyde 1925–2014

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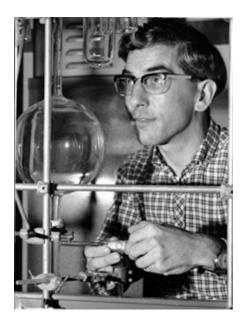
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Bruce Hyde made seminal contributions to modern solid state chemistry, in particular to the understanding and characterization of non-stoichiometry and structural complexity in the solid state. His work showed unequivocally that non-stoichiometric crystalline materials were often much more highly ordered than previously believed, that the 'point defects' of conventional wisdom were in fact ordered into extended defects and that these defects were themselves ordered into structures of complexities hitherto unimagined. His deep understanding of crystal chemistry and structural relationships is apparent in his two co-authored books with his closest colleagues, Sten Andersson and Michael O'Keeffe. It also led to his mentoring an entire generation of younger Australian (and international) solid state chemists.

#### A Welsh Childhood (1925–34)

Bruce Godfrey Hyde was born to his beloved Mam, Emmy (Beatrice Emily Godfrey), and father Charles on 13 March 1925 in their council house in Abercynon, a typical mining town, in the blackened Cynon valley of south Wales, and died in Canberra, Australia on 16 February 2014 aged 88. His father, Charles, was down in the pits by 14 years of age, digging coal and, like his father before him, working on the railways by 16. Though forced to work long hours, and often away from home, a railways job protected the family from the extremes of poverty then rife in Wales. Welshmen from the Valleys like Charles were hungry for education, but trapped in their class by birth. A sickly but bright child, Charles was unable to take up a local school scholarship for lack of money. As Hyde wrote 60-odd years later: 'Without education, ...there were really only two options-the railways for the more fortunate, the pits for the less fortunate.<sup>1</sup>

The deprivations of his own youth left Charles a strict father, determined to push his children. Emmy was less ambitious for her children, and gentler and more accepting of her station in life. At the end of his life, Hyde recalled her steady presence in his childhood. Home life was dominated by the fierce strictures of Chapel, leaving



little room for fun or moral latitude. Music and poetry, deeply embedded in Welsh culture, provided a strong and less stern undercurrent to life and Hyde developed a deep love for music, singing in Welsh Chapel and Eisteddfodau choirs as a child, and later playing organ and piano for pleasure as a young man. Classical music was often played at high volume later in life. Thanks to travel as an adult, his musical loves extended to flamenco and American spirituals.

By the time he was four, Hyde was attending the Abertaf Primary School. He was pushed hard from a very early age by his father, a pressure that he came to believe left him lacking in confidence for decades. His childhood marked him for life: fiercely proud of Welsh and Celtic history and culture, unashamedly on the side of the working classes and instinctively suspicious of English authority, 'manners' and social pretence. Like his own father, he also harboured the introspective, darker and sterner aspects associated with the Welsh character. His best friend was Irving Donovan, from a big Irish family, who lived down the street in a relatively grotty house. He loved their wild Irishness. Later in life, his closest professional friendships were also with relative outsiders, with whom he instinctively felt more connected.

#### Devon (1934-43)

'Abercynon ...set my course for life',<sup>1</sup> he mused in his eighties. Aged nine, the family left the Welsh valleys, moving to the small town of Newton Abbot, in rural Devon, England, where his father was transferred on the Great Western Railway. At first he attended the local All Saints Primary School; within a few months he 'sat the scholarship' to allow him to attend the Newton Abbot Grammar School fee-free. Though now living in a larger (council) house, the family could not afford school fees. Access to education relied on success in the exam. Thanks to a scholarship he spent the next six years at the local Grammar School, passing his School Certificate at 15 years of age.

In 1941, Hyde started his first job, at the Newton Abbot gasworks as assistant to the chemical analyst, responsible for ensuring the purity and safety of the town gas. To his amazement, within six weeks, his boss was gone and he was now running the chemical analysis laboratory! The chemistry that he had hated learning at school was suddenly real and practical, and he loved it. Aged just 17, and with the encouragement of the gasworks management who recognized his practical skills, he had the Newton Abbot analytical chemistry laboratory refurbished to his own specifications. In addition to testing the town gas for calorific value, as well as levels of coal gas, hydrogen sulfide, oils and ammoniacal liquors, he was officially responsible for extracting benzol from the gas to use as petrol, in short supply due to the war.

# The Navy (1943-7)

Once 18, he was called up to serve in the armed forces and his gasworks idyll was over. He joined the Navy, from where he went straight in to the Fleet Air Arm, thanks to his technical skills. His actual war training was minimal: largely consisting of just one day on a rifle range in Cheshire, 'where they tried to teach us to shoot a 0.303 rifle without shooting each other.' The next four years were spent away from home and family. After training in England and Scotland, he left England a qualified Air Radio Mechanic, and boarded the Stirling Castle in Liverpool in 1945, heading for New Zealand and Australia via the Panama Canal. Though the European war was over, Japan remained at war, and the Fleet Air Arm was sent to the Pacific theatre where its task was to set up Mobile Operational Air Bases in the Pacific.

On board he was suddenly exposed to another world: he saw his first 'dark-skinned' people-Maori ex-POWs, returning home after years in German camps who sang their songs, accompanied by swaying guitars each night in the ship's mess. Many Australian ex-POWs were also aboard, finally going home after five years in German camps. Australia was a revelation. 'We blossomed', he wrote later, 'and opened up in the sun and freedom of living in Australia, and became different (and nicer and cleaner) people.' From Sydney, he was sent to Queensland, where he and his naval mates hung about. They were no longer needed to fight the Japanese, who had surrendered. They hitch-hiked into the Queensland interior around Okie (southern Queensland), where he fell in love with the Australian bush.

He was eventually posted to *HMSImplacable*, a huge aircraft carrier stationed in Sydney but due to pay a goodwill visit to Melbourne. Despite intense stomach pain for a week, he crawled on board, determined to go. The next morning, acute appendicitis was diagnosed and he was carted off to hospital in Sydney in an ambulance. He was visited in his bed by the Rear Admiral Surgeon, a clear indication that he was seriously ill. A telegram was sent to Newton Abbot informing his Mam and Dad. He was operated on, inexpertly: 'I had to be cut open again so that it could be restitched. I was ... apparently 'out'-helpless [unable to speak] but still feeling and [in fact] awake. They were about to cut when one of the attendants opened my eye and I heard him say 'He's not out yet'. What relief! They then put me out.' He celebrated his 21st birthday in the recovery ward. His hospital stay left him with a life-long fear of medical intervention, despite its having saved his life. A few weeks later, now fully recovered, he was once again aboard HMS Implacable, bound for England after first dumping US aircraft in the ocean off Sydney (Britain had to either pay for them or dispose of them with the end of the war, and lacked the means to do the former). It was a reluctant departure, and he was only persuaded to return home after his new Queensland friends convinced him to first gain some higher education in the UK before choosing a life in Australia.

Once back in the northern hemisphere, he remained in the Navy for another year or so, with little to do. Of that period, he learnt just two things: 'the corruption of the real service way of life ...and the joys of cheap pipe tobacco (1/– a pound)'. From then on he was rarely seen without a pipe in his mouth or close at hand. In his final years he resorted to importing his beloved *Three Nuns* tobacco directly from Switzerland, thanks to the virtual extinction of pipe smoking in Australia. In February 1947, he was officially demobbed, after nearly four years in the Navy. In the few months before starting studies, he assessed those years as follows:

Normally such an experience would be described as 'sacrificing the best years of one's life', but ...now I was fixed up to commence a science (Chemistry) degree at Bristol University ...The opportunity to go to University would not have arisen if there had been no war or call-up!

#### Bristol (1947–54)

Thanks to his pre-war, gas-works experience at home in Devon, Hyde was hired as a chemist at the Bedminster (Bristol) non-ferrous extraction factory of Capper Pass & Sons. There he quickly mastered the procedures for analysis of tin, bismuth and antimony contents, relishing working with 'real industrial chemists'. Capper Pass intersected in unexpected ways with Hyde's own life. An early eponymous family member was convicted at the Bristol Quarter Sessions for handling stolen metal and transported to Australia in 1819. Nevertheless, the family company subsequently thrived and later established the Alfred Capper Pass Chair of Chemistry in Bristol University.

In the summer of 1947, while out cycling through the West Country, Hyde met Jean Harvey, the sister-in-law of one of his shipmates on *HMS Implacable*. Love blossomed quickly and within a year, at the end of his first year of University studies in Bristol, they married. Jean was a *bona fide* Cockney, whose family home had been bombed during the Blitz, and she had resettled in Devon, just a few miles from Newton Abbot. Their union was a happy one, and produced three children, but was tragically cut short by Jean's death shortly after childbirth just seven years later.

Undergraduate university life was a revelation. While school had been a struggle, his physics, chemistry and mathematics studies in Bristol were a pleasure. Now matured by years in the navy, no longer constrained by working life or his ever-watchful father, his enthusiasm for science grew strongly. His strong practical background in chemistry afforded him a running start. The mysteries of physics too suddenly seemed to clarify, likely helped by his own Radio Mechanics training in the Royal Navy. His laboratory books from those undergraduate years are an extraordinary testament to how seriously he grasped his new opportunities: they reveal an extreme level of competence in experimental procedure, and an almost obsessive level of meticulousness in his scientific drawings. That skill was to serve him well in later years, when so much of his time was taken up with painstaking hand-drawn diagrams of crystal structures.

At the end of the third year of his undergraduate degree (1950) his first child, Ian, was born. A happy family life brought new-found stability and joy to his modest new world. Fatherhood was to be a huge part of his life for many years to come. Perhaps, due to his changed circumstances, after excelling in the first three years, he failed to gain a coveted first-class Honours degree, and was awarded BSc Hons (IIa). Nevertheless, the experimental project associated with his Honours year, one that required delicate measurements of the vapour pressures of sucrose solutions, fed his growing enthusiasm for laboratory research.

Whereas the Second World War allowed him to enter a university, the generosity of the City of London's Worshipful Co. of Haberdashers allowed him to remain there, part-funding his PhD studies for the next three years, thanks to his wife Jean's Cockney heritage. The balance of his income during that period came from additional grants from equally obscure charities, as well as earnings from teaching chemistry at nightschool. The thermodynamics of sucrose solutions were again the focus of his PhD program, focused on accurate measurements of heats of solution, under the supervision of W.J. Dunning. Academically, he was brought up in an era when solid state chemistry, at least in the Englishspeaking world, had only existed in the academic curriculum for a decade or so and was dominated by kinetic and thermodynamic, rather than direct structural, studies. By contrast, continental (including Swedish) inorganic chemistry at the time, and since, has long had a strong bias towards structural chemistry. On the completion of his PhD experimental work in 1954, Hyde was a card-carrying thermodynamicist, whose experimental skills were virtually orthogonal to the structural studies of the solid state that eventually earned him broader renown. Events (see below) then transpired to prevent submission of his PhD thesis for another seven years, until March 1961, more than a decade after its commencement. Already, traces of his later conversion from thermodynamics to structural chemistry (or, as he later put it 'from physical chemistry into crystal chemistry'<sup>2</sup> can be detected in his thesis.

# The Melbourne University Period (1954–60)

After PhD studies, he was planning to take up a junior post at Alfred University, New York.At the same time, however, the eminent inorganic chemist J. S. Anderson visited Bristol, before his departure from Harwell to return to Melbourne University as Head of Department and Professor of Chemistry. Anderson was looking for postdocs to bring with him and Hyde accepted Anderson's offer to join him in Melbourne with alacrity, attracted by both Anderson's scholarship and the prospect of reacquainting himself with the Australian bush.

In order to save funds, Anderson arranged for the family to emigrate to Australia as '£10 Poms'. So on 18 June 1954, he set sail for Australia, accompanied by his two young children (Ian, then four and his two-year old daughter, Gwynne) and Jean, now heavily pregnant with their third child, Evan. Thanks to engine troubles, the trip was slow and unpleasant, especially for Jean. They finally arrived in Melbourne in July, where Hyde, along with Kevin Gallagher, also hired from England, commenced the first ever postdoctoral positions in the Chemistry Department at Melbourne University with Anderson. They rapidly set to work, and built their own vacuum systems and other labware.

Within months, tragedy struck. Just two weeks after Evan was born in late September, Jean died, leaving the young family utterly rudderless. 'Now I really was devastated', Hyde wrote later, 'I just couldn't believe or comprehend it. When I returned to the Department, I knocked on J. S.'s door, opened it, and said, 'She's gone'. 'My dear chap'he said, and told me to take off as much time as I wished.'Not long thereafter, some light shone into this darkness, thanks to his younger sister Norah's arrival. At just 19 years of age, she left England for Melbourne to care for three children under five. His colleagues helped Hyde through those difficult times in many ways. Just before Christmas, they arranged for him to leave his office and he returned to find it filled with gifts for the young children. Colin Sachs, the laboratory manager, took the infant Evan into his family home for some weeks, an act that reinforced Hyde's deep respect for the generosity of Australians.

One evening in June 1955, still dazed and saddened, Hyde accepted an invitation from Kevin Gallagher to go and have a glass of wine or two around the corner at Jimmy Watsons Wine Bar, in Carlton. There he met Marie Brady, a meeting that he later described as 'the supreme turning point in my life'. Within a year, they were married. And so to Hyde's Welsh sensibility was added Marie's Irish roots, strong and alive despite her pedigree as a third generation Australian.

J. S. Anderson's research program at Melbourne was focused on defect thermodynamics in non-stiochiometric compounds. Hyde was charged with continuing electrical measurements of PbS films in the presence of oxygen. The research was painstaking and slow. During this period, he became lifelong friends with Gallagher (who went on to University College, Swansea) and his wife Gislinde and a third postdoc who arrived shortly after Hyde and Gallagher, Derek Klemperer (who later joined Chemistry at Bristol). Together the group discovered the extra-curricular pleasures of Melbourne, with a shared love of classical music that they indulged thanks to the 'Soirées Musicales' of the Chamber Music Society. More prosaically, the group were introduced to the delights of the Victoria Market and pub counter-lunches by a department member, Arthur Palmer.

Despite many years of effort up to mid-1960, after the departure of Anderson from Melbourne, results from the work on PbS were inconclusive. In spite of the lack of publishable data (over a period that would today surely result in nonrenewal of a position), Hyde accrued valuable skills in working with gas absorption in oxides that bore fruit in the next phase of his career. Discouraging though this must have been, new horizons appeared in 1960 that altered his scientific trajectory over the ensuing decade. Slowly but surely, Hyde's explorations of the solid state shifted from quantitative physical chemistry and thermodynamic studies to an alternative, structural perspective of solid state reactivity and solid solutions.

That transformation was seeded in 1960 by the extended presence of Leroy Eyring, visiting Melbourne University from Iowa State University, and Sten Andersson, from the National Defence Research Laboratories in Stockholm, visiting the brilliant structural chemist David Wadsley at Fishermans Bend, Melbourne (CSIRO Minerals). Both had strong interests in non-stoichiometric solid solutions of apparent continuously variable composition and the mechanism for accommodating it. J.S. Anderson was at that time the leading proponent of a disordered, point-defect model for nonstoichiometry, based firmly on statistical thermodynamics. On the other hand, both Swedish (Magnéli and his group) and Australian structural chemists, notably Wadsley with whom Sten Andersson was then working very closely, argued strongly for a description in terms of homologous series of perfectly ordered phases whose

closely related structures could be determined by careful crystallographic methods. Though Andersson spent some time with the now leaderless group at Melbourne Chemistry (Anderson having returned to Britain), most of that year was spent working with Wadsley. The collaboration resulted in a series of structural papers that revolutionised the then understanding of 'nonstoichiometry', and demonstrated the central role of structure over thermodynamics.

Hyde began his career in J. S. Anderson's thermodynamic orbit, but finished it firmly ensconced within the crystallographic arena. From today's perspective, the then heated differences between the structural chemist Wadsley and the English FRS and founding member of the Australian Academy of Science (AAS), J. S. Anderson, during the 1960s may appear insignificant. In the early sixties, however, the philosophical differences between Anderson and Wadsley (and Sten Andersson) were sharp. In 1962, Wadsley wrote to Andersson of a joint book chapter: 'It is all structures, ... no ridiculous thermodynamics etc.'.3 In the opposite corner, J. S. Anderson FRS FAA wrote to Hyde in 1965: 'I would ... be glad to see Wadsley beginning to think again as a chemist rather than purely as a structural crystallographer'.

Anderson recognized the relevance of Wadsley's ideas in explaining compounds of 'grotesque stoichiometries' (Wadsley's phrase), with references to Wadsley's early work, in the third (1960) edition of the popular text by (Emeléus and) Anderson, Modern Aspects of Inorganic Chemistry. However, Anderson defended his belief in statistical disorder in the face of increasing evidence to the contrary, and Hyde was caught between these poles until the late 1960s. Post-retirement, Hyde deemed the tussle between these views to be sufficiently interesting and scientifically important to warrant his labouring for many years on and off on a definitive history of the case.<sup>4</sup> Historical judgements aside, the debate seeded more than a decade of research by Hyde and his colleagues in Melbourne, Iowa City, Phoenix, Sendai and Perth.

#### Iowa, Bristol and Arizona (1960-4)

As a result of Eyring's visit to Melbourne, Hyde spent a sabbatical during 1960–1 as Visiting Lecturer at Iowa, during which time they became firm friends. This period saw the abandonment of electrical measurements to probe solid state phases, replaced by precise isobaric measurements that allowed accurate maps of the temperature-pressure phase behaviour of oxides in the presence of gaseous oxygen. Their scientific collaboration resulted in Hyde's first paper as well as several other papers exploring the phase behaviour of multi-valent rare-earth oxides,  $RO_{2-x}$ , R = Pr, Tb and Ce. Earlier problems with trace contaminants were overcome, resulting in a series of phase diagrams, whose interpretation required the adoption of the notion of extended, organised 'defects' from lines to planes. The older picture of (spatially random) thermodynamic point defects of these sub- and super-stoichiometric oxides failed to explain these new, accurate, phase data.

Following the stay in Iowa, Hyde and his growing family, now with six children, flew back to Bristol, where he spent some months finalising his PhD thesis. The thesis was duly submitted and his PhD formally awarded. Despite being once more close to his parents and siblings in Devon, with his newly awarded PhD in hand, he was happy to leave England's shores once again, this time sure that he 'wasn't aPom any more'.

This time, he took the family to Arizona, following Eyring, who had just been appointed Chair of the Chemistry Department of Arizona State University. After a long drive through the USA, from Chicago to Arizona, they arrived in Tempe in late January 1962 and quickly settled in. Hyde's work had now moved a long way from his PhD studies of solvent-water effects. Nevertheless, he was scheduled to deliver a seminar on the work, entitled 'Sweet water' 20 months later, on 22 November 1963, in Phoenix, Arizona. The talk was cancelled, however, for this was the day President John F. Kennedy was assassinated in

Dallas, a thousand miles east. The nation was rocked, as were the Hydes by the cutting down of their hero.

In total, Hyde spent three years at Arizona. For most of that time, he continued the Iowa work on the complex rare-earth metal oxide systems, together with Eyring, Jim Sawyer and Judge Bevan (a visiting chemist on sabbatical from the University of Western Australia, UWA), culminating in a detailed thermodynamic study of the complex phase behaviour of the  $PrO_{2-x} + O_2$  system.<sup>5</sup> Bevan was a chemistry graduate of

Melbourne University, who had worked with J. S. Anderson in Melbourne and Harwell. Hyde and Bevan became close friends from this time together in Arizona. Their paths crossed often, scientifically and socially, up to their deaths within a few months of each other in 2014. A measure of their closeness can be gauged by Hyde's last chemistry research talk: delivered at the *Bevan Fest* (in Ballarat, 1995) of which Hyde was aco-organizer.<sup>6</sup>

Towards the end of his time at Arizona, Michael O'Keeffe joined the Department as Assistant Professor. Hyde and O'Keeffe, with a common background as Bristol University graduates, discussed research informally. Their friendship later grew into a scientific collaboration and close personal friendship that produced some of Hyde's most significant work.

#### The UWA Period (1964–75)

The uncertainties of an untenured post led Hyde, in 1964, to seek a more stable situation and with much encouragement from Bevan he won a position as Senior Lecturer (later Reader and Professor) in the Chemistry Department at UWA. The decision to leave Arizona was not an easy one, given the strong friendships established at ASU and joint research interests with Eyring. Nonetheless in December 1964, the entire family, including the nine children, born in Bristol, Melbourne, Iowa and Mesa, boarded S. S. Monterey for the crossing to Sydney. The family arrived in Perth, mid-January 1965, by train: two carriage loads of Hydes' emerged, including a six week old baby (Andrew), to be met by Chemistry staff with three large cars and a utility to take them to their new three-bedroom university home in Nedlands.

Immediately on arrival at UWA, Hyde set to work building a laboratory with the full support of Professor Noel Bayliss FAA, the eminent spectroscopist who was then Head of Chemistry. With considerable funding from the US Office of Air Force Research (the portrait photograph on the first page was published on the front page of *The West Australian* 3 December 1965 and shows Bruce Hyde hard at work in his laboratory at the University of Western Australia. The page one story celebrated the award to Hyde of a research grant from the US Air Force), he bought much high temperature thermobalance equipment, including Ainsworth and Cahn microbalances (set up by Frank Lincoln, a postdoc and later lecturer at UWA, and Dick Merritt, a PhD student) to continue his detailed thermodynamic studies of oxides. Hyde was a superb (and tenacious) experimental scientist. For example, in 1968 he bought precisely six milligrams of highly radioactive CmO<sub>2</sub>, at great expense (~\$4000), in order to conduct tensimetric experiments on the system. Then, with the help of Theron Felmlee (aUS postdoc) and Lincoln, he installed a Cahn microbalance and built a vacuum line in a glove box in the 'hot' Atomic Energy Commission laboratories at Lucas Heights in Sydney, to carry out the experiments.

Hyde's time in Arizona had matured him scientifically and exposed him to the crystallographic understanding of oxides, thanks to the presence there of Bosse Holmberg (a close colleague of Sten Andersson, then on sabbatical from Stockholm) as well an increasing familiarity with the structural work of Magnéli, Wadsley and Andersson. Just before leaving Arizona, he wrote an excited letter to J.S. Anderson, outlining a dislocation growth mechanism to effect Wadsley and Andersson's 'crystallographic shear' (CS) operations. The result was a series of papers on ordered and disordered CS structures, published after Hyde reached Australia, the first in 1965 and the last and most detailed in 1967.<sup>7</sup> They showed his growing interest in structural chemistry and his entry into the debate regarding the structural mechanisms underlying non-stoichiometry.

At the time, it was becoming clear that many 'non-stoichiometric', solid solution phases actually contained complex but closely related, ordered phases of fixed stoichiometries and that the structures of many of these complex compounds could be explained in terms of regularly spaced CS planes. Hyde had the insight that non-stoichiometric materials at even higher temperatures might have CS planes in disordered fashion that could explain the classical 'solid solutions' of wide compositional range at the highest temperatures without the need for any point defects at all.

To his great good fortune, during his first full year in Perth (1965), Melbourne hosted an international conference involving twin symposia, 'Electron Diffraction' and 'The Nature of Defects in Crystals', co-organized by the International Union of Crystallography and the Australian Academy of Science. The latter was explicitly designed to showcase the new structural ideas from Wadsley's camp along with the more traditional views of statistical thermodynamicists. Hyde presented two papers to the 'Defect' symposium, one covering his Arizona experimental work and the other his theoretical ideas on dislocation mechanisms and the formation of shear structures. Sten Andersson stopped in Perth en route to the meeting to discuss the latter. Within a couple of years they became semi-regular correspondents and clear friends.

The 1965 Melbourne meeting (and a local follow-up meeting organized by Hyde and Bevan at UWA in 1966) highlighted the increasing power of transmission electron microscopy (TEM) in exploring solid state structures *and* defects therein. The technique had matured substantially since the pioneering work of Ruskain the 1930s, thanks to theoretical and instrumental developments spearheaded largely by Cowley and Moodie (also at CSIRO in Melbourne). It was rapidly becoming clear that TEM was well on the road to imaging extended defect ordering in crystals. To quote Hyde, after the meeting it was clear that 'every solid-state chemist worth his salt *had* to get into EM work'.<sup>8</sup>

Only two years later, in 1968, Wadsley and the pioneering electron microscopists John Allpress and John Sanders FAA (at CSIRO Tribophysics, in Melbourne) published the first relevant lattice imaging TEM paper to provide definitive evidence of the existence of CS planes and block structures<sup>9</sup> in high temperature Nb<sub>2</sub>O<sub>5</sub> and related tungsten niobates, materials where thermodynamicists had earlier expected point defects to predominate. In a characteristically feisty letter to Andersson around March 1968, Wadsley wrote

The presence of Schottky-Wagner [point] defects is the last hope for these people ...Allpress and I will finally stop all this nonsense ... We had a session with Amelinckx [who was visiting Wadsley at the time], Hyde ..., Allpress, Sanders and myself ...out came Allpress with all his stuff ...they finally agreed that this random shear plane stuff is correct ...Hyde said that ...even P-T-X stuff is not precise enough in these systems ...But these people are all pretty slippery<sup>10</sup>



**Figure 1.** An animated discussion of CS planes in reduced  $Cr_2Ti_{n-2}O_{2n-1}$  rutile (during the time of the 1971 NBS symposium on Solid state Chemistry held at Gaithersburg, Maryland) requiring the consumption of much beer and the use of copious golf balls and ping-pong balls involving Bruce Hyde (foreground on the right in both), Mike O'Keeffe (on the right, in the left hand photo), Sten Andersson (back right in both photos), Jean Galy (back left in both photos) and Les Bursill (left foreground on the right), with Bob Roth taking the photos. In the improvised model, golf balls represent Ti<sup>4+</sup> ions, ping-pong balls Cr<sup>3+</sup> ions and aluminium cans (minus the labels) the O<sup>2-</sup> ions. (Photographs courtesy of Robert S. Roth.)

Slippery though he may have appeared to Wadsley, Hyde was converted. Not much later, he hung a framed photographic portrait of Wadsley on his office wall, which was to be found in all successive offices until his retirement, after which he hung it on his wall at home. Along with many colleagues, he considered Wadsley's untimely death in early 1969, aged only 50, a tragedy for solid state chemistry.

Given the beautiful early TEM results,<sup>11</sup> Hyde leapt at the chance to explore such systems himself. He gained access to a TEM at UWA, and initial results with postdocs and Research Fellows (including with Dick Tilley from Oxford) encouraged him to request funding for his own machine, a novel and expensive request from a chemist. Thanks to support from Bayliss, the request was funded and he took up microscopy in earnest. In 1968, he spent two months in Sendai with Den Watanabe, a leading electron microscopist and Osamu Terasaki, then a student (and later a world leader in high resolution electron microscopy, HREM) and exciting results followed. In Sendai, Terasaki and Hyde explored sub-stoichiometric TiO<sub>2-x</sub> oxides with grotesque stoichiometries of the form  $\text{Ti}_n \text{O}_{2n-1}$ ,  $15 \le n \le 36$ , which nonetheless were highly ordered.

The work was continued back in Perth, with the help of the young Australian microscopist

Les Bursill (a postdoc and recent postgraduate from Alex McLaren's laboratory at Monash University, who later also became a renowned HREM expert) and Don Philp (a PhD student). This led to a seminal joint paper<sup>12</sup> (in 1969) on the direct observation of extended defects in slightly reduced TiO<sub>2-x</sub> and meant that elaborate thermodynamic treatments in terms of point defects in non-stoichiometric compounds of this sort were meaningless. Nevertheless, there was still considerable opposition, at the time, to such ideas. But in an exquisite series of papers on reduced rutiles from 1969–73,<sup>13</sup> Bursill, Hyde and Philp showed that not only did disordered CS planes exist but that under the right conditions they could even change orientation (the later famous 'swinging shear planes') to give an ordered structure of virtually arbitrary composition. Such classic work showed unequivocally that 'non-stoichiometric' solids were much more highly ordered than previously believed (Fig. 1). The 'point defects' of conventional wisdom were in fact ordered into extended planar defects, and these defects themselves ordered into structures of a complexity hitherto unimagined. Much of this work was done by TEM and Hyde was among the first to recognize the potential it had to solve problems in solid state chemistry. His groups at Perth and later Canberra were surely among the first to set up TEM in a chemistry

department. The TEM had become a laboratory for inorganic solid state chemistry and Australian science was now very much on the world stage. Importantly, the thermodynamicists capitulated (at least as far as grotesque non-stoichiometry was concerned) and J. S. Anderson took up electron microscopy.

Hyde was now among the vanguard of the new structural approach to solid state his closest collaborators were Sten Andersson and Michael O'Keeffe. Both collaborations were to continue for decades, and their joint research and publications together were his proudest scientific achievements. From these very productive collaborations emerged some 24 journal publications in total as well as two highly valued books on inorganic crystal structures: a research monograph *Inorganic Crystal Structures* (begun in Leiden and published in 1989) and an excellent introductory text, *Crystal Structures: 1: Patterns and Symmetry*<sup>9</sup>.

Hyde's work with O'Keeffe and Andersson was focused on structure building principles, i.e. on new ways of describing structure types and their relations as well as transformation mechanisms from one to the other. The construction of elaborate and often beautiful 3-dimensional polyhedral models,<sup>15</sup> designed to highlight these structure building principles, were later a feature of all their offices. As a result, Hyde acquired an almost encyclopaedic knowledge of crystal structure types and crystal chemistry. This is illustrated most particularly by his book with Andersson.<sup>16</sup> Their work initially focused on extending Wadsley and Andersson's original CS concept to new types of planar faulting, but then moved on to other ways of accommodating non-stoichiometry (such as regular chemical twinning of well known structure types on the unit cell level as a structure building entity, see e.g. Figure 2, or rotation faulting enabling e.g. the  $ReO_3$  structure type to be related to a host of other structure types such as the 'bronze' and 'tunnel' structures<sup>17</sup> etc.) These were novel and elegant approaches that enabled very many structure types to be understood and related to one other.

In 1974, Andersson, Hyde and O'Keeffe teamed up with Tony Bagshaw (a postdoc from Oxford, later a lecturer at UWA) to publish a definitive account of ordered defects in the solid state in a characteristically comprehensive 50

**Figure 2.** A polyhedral model of  $MnY_2S_4$  involving regular chemical twinning of the octahedral *NaCl* structure type on the unit cell level, giving rise to trigonal prismatic coordination polyedra at the twin interfaces. (Photograph courtesy of Bethany McBride of the Functional Materials Laboratory, Research School of Chemistry, The Australian NationalUniversity.)

page review.<sup>18</sup> That work marked, more or less, the closure of the first fruitful phase of Hyde's research career, dedicated principally to understanding the nature of extended defects in oxides and related materials and the development of real crystal chemistry.

# Leiden Period (1975-7)

At a personal level, Hyde's research was taking him more frequently to mainland Europe. He liked its culture and respect for art, classical music and scholarship. Thus, in 1975, after more than two years of negotiations, he accepted the Chair of Inorganic Chemistry at Leiden University, in the Netherlands. Aged fifty, he departed from Perth for Leiden, with his wife Marie and the younger complement of his offspring. The departure coincided with his election as a Fellow of the AAS. Though determined to make a definitive break with his academic home for the previous decade, he prudently sought leave from UWA, so that the move was not irreversible.

As it transpired, the cultural shift was difficult for both Bruce and Marie. In the aftermath of late sixties' radicalism on campuses worldwide, Dutch universities adopted democracy to (in Hyde's view) an unworkable extreme, which hindered both significant departmental reform and research activity. At home, Marie's life proved to be unbearably isolated and she insisted they return to Australia. Thus, only two years after leaving Perth, he returned to UWA, happy to be back in the sunshine but somewhat sad to have left Europe behind.

His time in Leiden was, nonetheless, productive. Kees Plug and Martin Bakker started PhD studies with Hyde while Bert Prodan arrived as a scientific co-worker. The proximity of Andersson in Lund (Sweden) and extended visits by O'Keeffe led to significant advances in their joint research. Hyde together with O'Keeffe explored tetrahedral and octahedral structures, in particular co-operative polyhedral rotation as a mechanism for structural transformation. This work led to more general exploration of crystal structures with O'Keeffe, including detailed studies of the importance of non-bonded cation-cation interactions and its structural consequences for a host of materials. This work challenged the prevailing orthodoxy of anion versus cation 'sizes'.

# The Second UWA Period (1977-8)

Hyde returned to UWA in early 1977, greeted by the award of the locally prestigious H. G. Smith Medal of the Royal Australian Chemical Institute. Within a year or so, the reality of a continued career in Perth was wearing thin, despite his renewed enthusiasm for the collegiality and decency of his colleagues at UWA. He was finding the teaching load, as well as the hot summers and intermittent, long-distance travel tiresome. Simultaneously, the Australian National University (ANU) in Canberra was keen to initiate a new group in Inorganic Chemistry at the then wellfunded Research School of Chemistry (RSC). After some hesitation, the lure of a research-only position, together with a promise of substantial set-up equipment grants and guaranteed funding for several junior researchers, was accepted. The decision was accompanied by a mixture of enthusiasm for a new start and sadness at leaving his long-term Perth colleagues, particularly Frank Lincoln, Gerry Bottomley, David Kepert and Graham Chandler.

And so, in January 1979, he made his final professional move, to Canberra, via a slow road trip with Marie and the three youngest children. They arrived in Canberra in the summer heat, and with the help of the only remnant of his Perth team, Jeff Sellar, immediately set to work.

#### The ANU Period (1979–90)

In his earlier years at Chemistry in Perth in the mid-to-late 1960s, Hyde's scientific output had suddenly jumped to a new level, thanks to the support of his peers there, as well as ample grant support to pursue his own research agenda. Similarly, in Canberra, his research activity once again ramped up to a new level, with the result that more than a half of his publications stem from the Canberra period. His final decade of scientific activity was thus an extremely fruitful one, and he grew to love Canberra for its clear air and sunny skies. The support of his senior colleagues, including Arthur Birch and David Craig, undoubtedly revivified his research batteries.

Though by its own lights now labouring under reduced circumstances, the RSC was still an international scientific crossroads with large numbers of visitors each year. To Hyde, one of its great attractions was the possibility of hiring young Research Fellows (RF's) with the skills required for a full-scale assault on 'crystal psychiatry', as he called it. The initial team of Research Fellows consisted of Joyce Wilkie, Jeff Sellar and Harry Nyman respectively. Unsurprisingly, the earliest large purchases were electron microscopes: the first, a JEOL 100CX workhorse instrument followed shortly thereafter by a JEOL 200CX (in January 1980). The 200 kV instrument boasted a then unheard of resolution of  $\sim$ 2.5 Å. Its chief drawback was an extremely delicate top-entry stage permitting a maximum specimen tilt of only  $\pm 10^{\circ}$ . Nonetheless, in those early ANU years the 200 CX was worked day and night, often with Hyde present, spurring the users on to discover instances of new crystal chemical principles he felt must underlie the behaviour of a whole range of materials. Of course, many samples had to be synthesized so a large furnacefilled laboratory for the preparation of specimens was also part of the set-up.

The earliest Visiting Fellows (VF's) were the Slovakian mineralogist Emil Makovicky from Copenhagen, the solid state chemist Koji Kosuge from Kyoto and, in 1982, J. S. Anderson, Hyde's old postdoctoral research leader (who had retired from Oxford to Canberra). Makovicky specialized in the structures of sulfides and complex sulfosalts, a topic strongly taken up later by many group members. Hyde was around this time working on a large and characteristically thorough review paper with Makovicky on the growing family of misfit (or vernier) layer sulfide compounds that he and his group were in the process of investigating experimentally.

Over the next years, Hyde surrounded himself with a veritable Foreign Legion of visitors, postdocs and RF's working on a broad array of complex, solid state chemical problems: often associated with mechanisms accommodating variable stoichiometry. Carlos Otero-Diaz arrived as a post-doc (later an RF) from Spain to work on non-stoichiometry in the  $Yb_{3-\delta}S_4$ and the 'LaCrS<sub>3</sub>' misfit layer structure in January 1980. He and Hyde discovered an incommensurate modulation in the former associated with Yb2+/vacancy ordering while the latter were later shown to be composite modulated structures involving continuously variable local co-ordination polyhedra. Otero-Diaz was soon followed by RF's Piers Smith, John Parise and Allan Pring (all working on heavy-metal sulfides and selenides; Smith and Pring were both experienced electron microscopists), Geoff Anstis, who strengthened greatly the group's image simulation capacity) and Lars Stenberg who worked on modulated structures in the alkaline earth silicates. Hyde was becoming increasingly interested in modulated structures and their characterisation via EM became a major research theme in Hyde's group.

Tim White was Hyde's first ANU PhD student, and worked on mineral systems. Jacques Barbier and Tim Williams were Hyde's second and third ANU graduate students. Barbier worked on spinelloid-related phases and polymorphism in Ca<sub>2</sub>SiO<sub>4</sub> while Tim Williams worked on complicated mineral structures, such as cylindrite, franckeite and cannizarite, composition  $\sim$ Pb<sub>46</sub>Bi<sub>54</sub>S<sub>127</sub>! There was also a flow of further VF's such as Kenji Hiraga from Japan (another expert electron microscopist) and a host of shorter-term visitors, both from Australia and

overseas, including Alex McLaren, Les Bursill and Peng Ju Lin, Alec Moodie FAA, Andy Johnson, Christer Svensson, Bob Roth, Jan-Olov Bovin, 'Terry' Willis, Gus Mumme, Jean Galy, Gerry Gibbs, Mike Murray FTSE and Ian Grey.

A rather solitary project over the same period absorbed a great deal of Hyde's time: the first drafts of his monograph with Sten Andersson.<sup>19</sup> It summarized much of what Hyde and his close colleagues had discovered and was dedicated 'To David Wadsley, who led the way'. While the book progressed, he conducted an RSC seminar series on crystal structures for the benefit of students, RF's, postdocs and visitors, a chapter at a time. For the uninitiated, the number and complexity of the structure types and Hyde's trance-like state when describing them presented an alarming prospect. The Old Hands were safe enough but more than one audience member of the balland-stick fraternity, on being quizzed about what he had gleaned from the seminar, found himself upbraided with the only-half-serious censure 'I've taught you everything I know and still you know nothing!', one of Hyde's more didactic coinages.

Hyde was particularly interested in mineralogy during this period and was determined to link up with geologically orientated crystallographers at the Research School of Earth Sciences (RSES): people like Ted Ringwood, John Fitz Gerald and L. G. 'John'Liu. (He always insisted that 'Pressure was THE variable!') In 1984-5, in conjunction with Alex MacLaren and RSES's Mervyn Paterson, he was instrumental in the setting up of a Mineralogy Research Centre involving the purchase of a new TEM. A vivid impression of the early Canberra years may be glimpsed in the review article<sup>20</sup> by Hyde and some of his colleagues in 1985 detailing TEM studies of oxides and sulfides in which the concepts of CS, double CS combined with rotation operations, structure and disorder in mineral systems through modulated structures, in confirmation of the programme carried by Hyde and his co-workers since the 1960s.

Another Hyde coinage was 'Landau Theory [introduced into physics by Lev Landau in an attempt to formulate a general theory of continuous phase transitions] is the last refuge of the Scoundrel'. He may have needed to trim sail somewhat in 1986 with the arrival of Ray Withers as a new RF John Thompson had also joined the group in 1985 and they continued the earlier work on modulated structures until the arrival of the high temperature superconductors in 1986. It was then all hands to the pump for a few hectic years, including Hyde himself, J. S. Anderson, Judge Bevan, Frank Lincoln, John Fitz Gerald, Mervyn Paterson, Andrew Stewart and Reine Wallenberg. The work was conducted with an intensity and excitement that compared with the earliest days of the Wadsley-Anderson battles,

though this time the competition was universal. The effort led to a flurry of papers but after a frenetic year or two, he decided to quit the race and return to their main research interests, principally modulated structures and wide range non-stoichiometric, solid solutions in the  $CeO_2$ - $Y_2O_3$  system.

Before the start of his retirement at the end of 1990, Bruce and Marie ventured once more to Europe, where they travelled and Bruce lectured widely, revisiting old friends and colleagues. Thanks to an extended stay in Madrid hosted by his erstwhile postdoc and friend Carlos Otero-Diaz, the trip was one that lived on in stories over the dinner table, often inspired by background cante jondo flamenco music.

At the end of 1990, Bruce Hyde retired quietly from ANU, followed by an informal meeting at UWA, consisting of two-and-a-half days of talks followed by a memorable dinner at University House, with family and colleagues from the Chemistry and Physics Departments at UWA, including Sir Noel Bayliss FAA, Frank Lincoln, Gerry Bottomley, David Kepert, Andy Johnson, Allan White, Ted Maslen FAA and Andy Johnson. Though he had worked ferociously throughout his research career, he gave it all up within a couple of years of his mandatory retirement aged 65. He accepted the change with little overt regret, happy to offer younger colleagues the space and opportunities he had enjoyed in his own scientific youth. He then spent some years exploring personal and scientific histories: that of Dave Wadsley and the success of crystallographic ideas over more classical statistical thermodynamics,<sup>2</sup> and then his Hyde ancestors, culminating in a substantial tome of family history,<sup>1</sup> finished in 1996.

In the manner of his generation, his every domestic need was attended to by his indefatigably loyal wife, Marie. So it came as a terrible blow when Marie died somewhat unexpectedly, aged 79 in 2011. Once again, he buried his wife, and the terrible sadness experienced by the loss of his first wife Jean 55 years earlier was revisited. Just one year before Marie's death, the family had gathered to bury their youngest son, Andrew, in Esperance. As a result of those losses, Hyde's final years were difficult ones, but he bore these hardships with characteristic stoicism. His final years were brightened by the presence of his daughter Melony and son Dewi, who moved to Canberra to care for him to his death. On the evening of 16 February 2014, he died peacefully at home, surrounded by many of his children, after refusing medical intervention earlier that morning.

# Endnotes

- <sup>1</sup> Unless otherwise noted, this quote and others throughout the paper are quotes from Hyde's personal notes (handwritten 1987–94) and 'Kin (and some kith)', his unpublished personal family history (c. 1996).
- <sup>2.</sup> B. G. Hyde, 'David Wadsley's Science' (unpublished, 2002, pp. viii+181 with 33 figures plus a 33 page appendix, Spp. = 222). A copy is held at the Black Mountain Library of the CSIRO in Canberra.

- <sup>4.</sup> As above.
- <sup>5.</sup> B. G. Hyde, D. J. M. Bevan and L. Eyring, 'On the Praseodymium + Oxygen System', *Philosophical Transactions of the Royal Society of London, A* 259 (1966), 583–614.
- <sup>6.</sup> 'Some Cubic (Pa3) Structures of Stoichiometry MX<sub>2</sub>', April 22, 1995. Among the speakers at the two day meeting were many eminent Australian solid-state scientists whose research overlapped with that of Hyde and Bevan, including Allan Pring, Peter Goodman, Ian Grey, Frank Lincoln, Alex McLaren, Ted Maslen, Alec Moodie, Richard Welberry and Ray Withers.
- <sup>7.</sup> J. S. Anderson and B. G. Hyde, 'On the Possible Role of Dislocations inGenerating Ordered and Disordered Shear Structures', *Journal of Physics and Chemistry of Solid,s* 28(8) (1967), 1393–1408.
- <sup>8.</sup> Already cited (n. 2).
- <sup>9.</sup> J. G.Allpress, J. V. Sanders and A. D. Wadsley, 'Electron Microscopy of High Temperature Nb<sub>2</sub>O<sub>5</sub> and Related Phases', *Physica Status Solidi*, 25 (1968), 541–550.
- Letter from Wadsley to Sten Andersson, 5 February 1968 and quoted in above (n. 2).
- <sup>11.</sup> L. A. Bursill, B. G. Hyde, O. Terasaki and D. Watanabe, 'On a New Family of Titanium Oxides and the Nature of Slightly-reduced Rutile', *Philosophical Magazine*, 20 (1969), 347–359. See also n. 9.
- <sup>12.</sup> As above.

<sup>&</sup>lt;sup>3.</sup> As above.

- <sup>13.</sup> See Supplementary Bibliography available online.
- <sup>14.</sup> As above.
- <sup>15.</sup> Manufactured and sold by 'Polyhedral Solids', run by Sten Anderssson from his Lund apartment.
- <sup>16.</sup> B. G. Hyde and S. Andersson, *Inorganic Crystal Structures* (New York, 1989).
- <sup>17.</sup> Already cited (n. 13).
- <sup>18</sup> B. G. Hyde, A. N. Bagshaw, S. Andersson and M. O'Keeffe: 'Some Defect Structures

in Crystalline Solids', *Annual Review of Materials Science*, 4 (1974), 43–92.

- <sup>19.</sup> Already cited (n. 15).
- <sup>20.</sup> J. Barbier, K. Hiraga, L. C. Otero-Diaz, T. J. White, T. B. Williams and B. G. Hyde, 'Electron Microscope Studies of Some Inorganic and Mineral Oxide and Sulphide Systems', *Ultramicroscopy*, 18(1–4) (1985), 211–234.