

SYSTEMATIC ASR EXPERTISE - AUSTRALIAN RESEARCH 1940'S TO 1958

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ABSTRACT

The Australian research for exploration of the nature of ASR from the early 1940's to 1958 was planned and managed by the Commonwealth Scientific and Industrial Research Organisation (CSIRO) as a major national research effort with H.E. Vivian as the leading operating research officer throughout the programme. Its emphasis was on gradual, systematic clarification of the interrelations between the chemistry of the reaction and the physico / mechanical effects in mortar and concrete. In the present review of the programme reference is made to contemporary and later research overseas, which is closely related to or complementary to the Australian work, and which enhances its outstanding realisation of a holistic concept of ASR.

In this respect the CSIRO accomplishments are superior to what has been achieved by much of the later, more pragmatic and fragmented research elsewhere. In terms of strategic planning, course of operations and output the Australian investment has therefore still in 1996 great merits as a guidance for the still required work for final settlement of the issue of ASR as a global threat to concrete durability.

Keywords: Alkali, Silica, Reaction, Research, History, Holism.

INTRODUCTION

The first of twenty six published articles to appear over the next eleven years about the Australian studies of ASR mentioned that Th.E. Stanton's discovery in California in 1940 engendered concern in the Commonwealth Scientific and Industrial Research Organisation (CSIRO). (Alderman et al. 1947). The American studies had identified opal as a primary reactive component in the aggregates of affected field concrete, and opal was a well known mineral in Australian geology. Volcanic rocks with glassy silica were subsequently found reactive in the West and Southwest states of the USA, and these rock types were also common in urban regions of Australia.

Apparently CSIRO established contact with Mr Stanton before World War II reached the Pacific, and launched a programme to explore the possible consequences of the new knowledge for the uses of concrete in Australia.

The purpose of the programme was: *"To investigate the probable incidence of cement-aggregate reaction in Australia, and to provide a basis on which more fundamental investigations of the mechanism of the reaction could be developed"*.

The latter half of this strategic goal setting shows a remarkable early perception by the CSIRO leadership of the importance of scientific clarification of the fundamental mechanisms of ASR.

The following decade of the research created a never later surpassed system of interactive studies after the initial classification of the national aggregate resources and cements. Exceptional operational skill, common sense and dedication characterise the design and utilisation of the experimental test series which were made to explore how the chemical reaction in aggregate particles might cause cracking and expansion in ambient, hardened cement paste.

PROGRAMME - PLANNING AND REALISATION

Samples of typical aggregate materials were collected from various parts of Australia a 7,686,000 sq. kilometre continent. Also samples of cement which covered the available range of alkali contents were collected. The aggregates were characterised geologically and mineralogically. The cement brands were classified by chemical analyses. Stanton's mortar bar expansion test was taken as model for the experimental studies.

Documentary progress reports were published as a series of articles and bulletins over the 1947-58 period, at that time an unusual long-term continuity of studies of one subject matter by a public research agency with many obligations. A brief survey of the research progress was presented in the journal of the American Concrete Institute, April 1950. (Alderman et al. 1950). Co-author H.E. Vivian became later on a frequent contributor to the ICAAR conferences and reminded patiently the new researchers from countries where ASR hesitatingly became recognized about the already available knowledge. (Vivian. 1975, 1976, 1981^{a, b, c}, 1983, 1986, 1992)

TABLE 1 enumerates the documentary CSIRO reports with classification as (1) overview, (2) chemistry of ASR and (3) mechanism of ASR, and with identification of the authors. As appears H.E. Vivian was the single author of fourteen of the reports and co-author of other eight. He was, in other words, indisputably the permanent, all round engaged generalist of the team.

OVERVIEW

The first progress report, (Alderman et al. 1947) describes the origin, rock types and mineralogy of 78 samples of different Australian aggregate materials and two control samples (reactive and non-reactive). Ordinary and low heat Portland cement were supplied by 10 cement companies and represented the range of occurring alkali contents. The reactivity of different blends of aggregates and cement was then examined by the mortar bar expansion test.

Secondary silicification, i.e. dissolution of silicates in rock types and resolidification of silica in and on rocks was known as a common phenomenon in the Australian landscapes, and in particular in the hot, central desert plains. (The reason for this particular disintegration of natural rocks and the effects on alkali-reactivity of concrete aggregates were not further pursued in the programme).

Delayed reaction, i.e. expansion of mortar bars, was found with rock types in which the reactive constituent was micro- or cryptocrystalline quartz. (Gaskin et al. 1955). Macrocrystalline quartz was found to be non-reactive.

TABLE 1: Survey of the CSIRO programme for Australian ASR research 1940's to 1958

| Progress Report | | Study Category | | | | Author | | | | | | |
|-----------------|------|----------------|-----------|-----------|-------------|---------------|-------------|------------|------------|--------------|--------------|--------------|
| No. | Year | Overview | Chemistry | Mechanism | Vivian H.E. | Alderman R.A. | Gaskin A.J. | Jones R.H. | Davis C.E. | McGowan J.K. | Benneth I.C. | Roberts J.A. |
| I | | x | | | • | • | • | • | | | | |
| II | | | x | x | • | | | | | | | |
| III | 1947 | | x | x | • | | | | | | | |
| IV | | | x | x | • | | | | | | | |
| V | | | | x | • | | | | | | | |
| VI | | | x | | • | | • | | | | | |
| VII | 1947 | | x | x | • | | | | | | | |
| VIII | 1948 | | (x) | x | • | | | | | | | |
| IX | | | x | x | • | | | • | | | | |
| X | | | x | x | • | | | | | | | |
| XI | | | x | x | • | | | | | | | |
| XII | 1950 | | x | x | • | | | | | | | |
| XIII | | | x | x | • | | | | | | | |
| XIV | | | x | x | • | | | | | | | |
| XV | | | x | x | • | | | | • | | | |
| XVI | | | x | | • | | | | | | | |
| XVII | 1951 | | x | x | • | | | | | | | |
| XVIII | | | x | | | | | | • | | | |
| XIX | 1951 | | | x | • | | | | | | | |
| XX | 1952 | | | x | • | | | | | • | | |
| XXI | 1955 | | x | x | • | | • | • | | | | |
| XXII | 1955 | | x | x | • | | | | | | • | |
| XXIII | 1955 | | | x | • | | | | | • | | |
| XXIV | 1955 | | | x | • | | | | | | | • |
| XXV | 1957 | | | x | | | | | • | | | |
| XXVI | 1958 | | x | x | | | | | • | | | |

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THE CHEMISTRY OF ASR

The programme did not comprise erudite contributions to the science of silica chemistry, but comprised applied chemistry to the extent it related to the physico / mechanical effects of the reaction. Opal was used as the preferred reactive aggregate, and the experimental test series comprised the measurements data, thorough observations of the visual effects of reaction, and intelligent deductions.

Some far reaching discoveries which sustained and complemented contemporary research accomplishments in the USA were elucidated:

- ASR is a chemical reaction by which the negative OH^- ion in the pore liquid of hardened cement paste in mortars and concrete converts silica in aggregate particles to a gel or sol along with absorption of water (i.e. the alkaline pore solution). (Vivian. 1950^{e, f}), (Davis 1951).
- The positive metal ion, Na^+ and K^+ cannot themselves cause the reaction, but different positive ions influence the cause and effects of the reaction differently. (Vivian. 1950^f, 1951^a)
- The pore liquid of hardened cement paste is an alkali hydroxide solution in which the solubility of calcium hydroxide is very low, and unable by itself to convert silica in aggregate particles to a gel or sol.
- The reaction product, alkali-silica gel or sol absorbs available water from the pore liquid or the ambient atmosphere. The alkali hydroxide concentration and the amount of water affect the rate of absorption and whether the conversion results in a gel or a sol. (Vivian. 1947^{a, b, c}, 1948, 1950^{a, b, c})
- The increase or decrease of the volume of the gel (or the amount of the sol) depends upon the amount of available water for absorption. (Vivian. 1947^c, 1950^f).
- The formation of gel as the reaction product creates a swelling pressure along with its absorption of water. Swelling pressure does not depend upon osmotic pressure by a solution in a semi-permeable membrane. (Vivian. 1947^c, 1950^f)
- The rate of absorption of water by the reaction product increased with increasing temperatures. (Vivian. 1951^b)
- The reaction takes place in reactive, siliceous aggregate particles. (Jones and Vivian. 1950), (Vivian 1950^{e, d}, 1951^c)
- Alkali hydroxide diffuses in the pore liquid from regions in the cement paste with high concentrations to regions with low concentrations, and from regions with no reactive to regions with reactive aggregates. (Vivian. 1947^a, 1948)
- The reaction is pozzolanic when the reactive material is present as finely ground particles (< 300 mesh). (Vivian. 1951^c) (Bennett & Vivian, 1955)
- The presence of CO_2 in concrete inhibits ASR. (Gaskin. 1947).

The authors of the progress articles referred to did - maybe unwittingly - reach much more than appreciated at the time of their work towards a universal concept of the chemistry of ASR. In any case, it is a logical inference emerging from their achievements that ASR is a hydration reaction, akin to the hydration of Portland cement. This perception was incidentally much later sustained by overseas researchers. (Farrouto and Haynes. 1973), (Idorn. 1980), (Regourd et al. 1981). Farrouto & Haynes demonstrated experimentally, and explained how different $\text{Na}_2\text{O} : \text{CaO} : \text{SiO}_2$

ratios determined whether the hydration caused the development of solid reaction products, exhibiting strength (high CaO concentrations) or no-strength, i.e. gel or sol products (high Na₂O concentrations), albeit not with constraints to make swelling of gel appear as expansive pressure. The perception of ASR as a hydration process is further sustained by the observation that the rate of the reaction increases with increase of the temperatures. This is indeed an early indication - corresponding to the contemporary development of the accelerated ASR test methods in the USA - that, being a hydration reaction ASR follows Arrhenius' law. This was much later experimentally confirmed elsewhere. (Tang Minshu and Han-Sufen. 1983)

The subtly designed and operated mortar bar test series which revealed that alkali hydroxide migrates in mortar bars, and hence tends to make the concentration uniform, was spectacular evidence of the chemical nature of ASR in concrete: The mobile alkali and hydroxide ions migrate in the pore liquid towards the immobile, solid reactive particles in which the chemical conversion happens.

This perception has two important implications:

- Uniform distribution of reactive particles in mortar bars with uniform alkali-hydroxide concentrations warrants high reproducibility of expansion test results. This has made it possible to adopt the "pessimum proportion" perception for laboratory studies of ASR.
- In field concrete with uneven distribution of reactive materials in aggregates alkali hydroxide is bound to diffuse towards high concentrations of reactive silica for corresponding high intensity of the reaction. This makes it impossible to adopt the "pessimum" principle for ASR in field concrete, and to transfer any empirical mortar bar test results to analyses or judgment of ASR in concrete.

The characterisation of ASR with finely ground reactive materials (opal) as a pozzolanic reaction which may prevent expansive ASR in concrete implies, that pozzolanic reaction generally must be perceived as primarily non-expansive, even beneficial ASR. This is indisputably sustained by the observation that the alkali hydroxide concentration in the pore liquid of cement paste prevails over the much less soluble calcium hydroxide. These Australian observations were in agreement with contemporary American research (Powers and Steinour. 1955^{a, b}) which relied much upon the achievements in the CSIRO programme. It is astonishing how difficult it has been since then to get this new infeasible perception of pozzolanic reaction as a beneficial modification of ASR broadly accepted against the classical imagination that pozzolans primarily react with calcium hydroxide in concrete. (Idorn. 1978, 1983), (Idorn and Roy. 1986).

THE MECHANISM OF ASR

The lasting value of the CSIRO team's study of ASR is augmented by the authors' ability to examine and deduct how the chemical reaction in aggregate particles may cause cracking and expansion in affected particles and ambient cement paste.

The mechanism was explained as follows: *"The development and widening of cracks by swelling particles are regarded as producing the expansion of mortar specimens. There is a positive correlation between the measured expansions of mortars*

and those calculated from estimates of the mean numbers and widths of cracks observed in mortar specimens". (McGowan and Vivian. 1952)

The test series in this late study report was the final documentation of their early discovery that "mortar expansion results from the swelling of reacted aggregate particles which cause cracking", (Jones and Vivian. 1950), which further confirmed earlier observations and deductions. (Vivian. 1947^{a, b, e}, 1950^{a, d, e}).

The exclusive use of Australian opal in all these test series made it possible to learn that the solid mineral substance was converted to swelling gel in reacting particles, or high concentrations of alkali hydroxide and abundant water might carry the reaction through to conversion of the mineral to an alkali-silica hydrate sol, which dissipated out in the pore volume of the ambient cement paste without causing swelling pressure, cracking and expansion. This clarification of the mechanism refuted effectively the elsewhere stipulated "osmotic pressure theory". (Jones and Vivian. 1950), (Vivian. 1950^f)

R.H. Jones and H.E. Vivian made microscopic examinations of thin sections prepared from mortar bars with ASR in their study of crack formation due to swelling pressure in reacting aggregate particles. (Jones and Vivian. 1950). They did not, however, produce suitable sections with harder materials than opal [The advanced thin section preparation by the epoxy-impregnation method, (Poulsen. 1958), made the closely following investigations in Denmark with opaline chert, dense flint and polymineral, reactive rocks complementary and utterly sustaining of the CSIRO studies. (Idorn. 1956, 1967).]

In the Australian thin sections dark, transparent zones were observed in the cement paste around reacted particles. (Jones and Vivian. 1950). The authors presumed - what has later on repeatedly been confirmed by other workers - that the darkening was due to depletion of crystalline $\text{Ca}(\text{OH})_2$ by its reaction with the alkali-silica gel or sol in the paste and in the cracks created by the swelling of particles. They also discerned isotropic material in cracks in cement paste and inferred with good reason that this substance was "the reaction product of alkalis and opal". These observations supported deductions made from earlier test results, (Vivian. 1947^b), that cracks around reacted aggregate particles "were partly filled with the reaction product". (In the thin section the isotropic material usually appeared as filling the cracks completely - maybe, though, due to the absence of third dimension observations in thin sections). Vivian showed also experimentally that the presence of calcium hydroxide in the reacting system of alkali hydroxide and opal produced a calcium compound with "little power to absorb water and swell, and very little strength or cohesion", (Vivian. 1950^f) and referred to "A calcium hydroxide-silica complex" in order to distinguish it from the "soluble sodium hydroxide-silica complex in the clear gel".

Thus, the several CSIRO approaches brought them close to the much later observations that alkali silica gel absorbs calcium hydroxide from ambient cement paste and becomes rigid, non-swelling, when it exudes out into the cracks in the cement paste which the swelling pressure in the particle has created. (Knudsen and Thaulow. 1975), and that uptake of calcium ions also happens in the rim zone of reacting opal particles, (Thaulow and Knudsen. 1975). These observations concurred with the Australian observations of the similar phenomenon in thin section examinations. (Jones and Vivian. 1950).

The mentioned Danish observations refuted the stipulation that alkali silica gel after its deposition in cracks in concrete might further swell and widen the cracks. (Jones and Vivian. 1950). This early conjecture was presumably an effect of the choice of opal as the modelling reactive material. ASR with opal was, like ASR with Danish opaline flint and glassy silica in volcanic rocks, Pyrex glass etc. profusely gel producing and thin sections might often show cracks apparently filled by gel. In contrast, ASR with dense flint and polymineral rocks with microcrystalline quartz, have been found to make expansive reaction happen with only minor gel production.

The patient, piece for piece building up of the concept of the mechanism of ASR was completed by series of measurements of the tensile strength of mortar bars in expansion test series. This approach established the distinction between the effects of swelling individually in reacting particles and the overall effects on mortar bars' tensile strength, vis-à-vis the measured expansions of bars. It was found that "*Serious reaction of aggregate in mortar which leads to expansion can be detected in a comparatively short time by tensile strength determinations. It is also possible that slow-reacting aggregates which are not easily detected by mortar bar measurements may be detected by this method. Observations indicate that fall in tensile strength always precedes measurable expansion*" (Vivian. 1947^c). See also Vivian 1947^{d, e}, 1950^{c, e}.

In the early 1950's the programme approached its termination, presumably because there were reasons for the CSIRO to change priority from a formidable investment in the ASR research to subject matters other than concrete. Three final studies were then made to relate the research to the actuality of ASR in field concrete, evidently realizing that investigations of the effects of ASR in existing structures in Australia - if occurring - would benefit from reasonable extrapolated deductions from the laboratory studies.

The effect of superincumbent load in mortar bar expansion was examined realizing that concrete in large masses in structures is submitted to imposed stresses, which normal mortar bar testing does not reproduce. (McGowan and Vivian. 1955). The concluding comments: "*Crack patterns are markedly affected by large loads. On the basis of the observations recorded in this paper it may be concluded that concrete structures undergoing expansion must develop a crack pattern that is governed by the restraints imposed by the design and magnitude of a structure. Examination of a vertical face of a large expanding concrete structure should reveal such features as: (i) fine, random surface cracks over the whole face; (ii) larger random deep cracks at the top of the structure, and (iii) well-spaced, open, vertical cracks which extend deeply into the structure. These latter vertical cracks may extend from top to base of the structure but should become less deeply penetrating towards the base. Crack planes parallel to the surface should also occur at various depths within the structure and it is the widening of these and their tendency to expand the concrete outwards that produce the large, open vertical cracks. These results also suggest that the whole mass of a large structure is not affected to the same extent by alkali-aggregate reaction. The probability emerges that the outer layer of concrete is cracked and expanded while the inner core remains relatively unexpanded. Since the cracks in the outer zone are interconnected, it should be possible by suitable recementing procedures to repair the structure and restore it to a condition approaching that of unaffected concrete. As a result of such restoration the weathering deterioration of damaged concrete could be checked or reduced--*" are admirably predictable for what field inspections in many

other countries have experienced with structures affected by harmful ASR. Implicitly their observations also suggest that cracking without expansion occurs in structures as well as in mortar bars.

The possibility to restore mortars which have cracked due to ASR by grout injection was examined. (Roberts and Vivian. 1955). Grouting with a Na_2SiO_3 solution appeared to be an applicable method; but trial in practice was not involved.

Series of experiments were made for comparison of the effects of expansive ASR in mortar bars and concrete, (Davis. 1957), apparently with the implications for larger masses of concrete in mind. The author found among other things that:

- A certain absolute minimum alkali content of the cement - 0.4% eqv. Na_2O - was necessary to obtain expansion in mortar bars. Increase of opal content required higher alkali content, and concrete bars needed more alkali for expansion than mortar.
- Large mortar bars expanded later than smaller ones, and developed the widest cracks.
- Concrete bars with inert coarse aggregate cracked and expanded later than mortars. The relationship was found to be:

$$T = \frac{t^2}{11}$$

where: T = time of significant expansion of a concrete bar
t = time of significant expansion of a mortar bar representing the mortar of the concrete.

- Expansion is a consequence of internal cracking due to swelling of reacting particles.
- Coarse, inert aggregate particles reinforce the concrete against cracking and expansion. Reinforcement bars do the same, however, over greater lengths and directional.
- Coarse, reactive aggregate particles ease or increase expansion, in particular with small amounts of alkalis. Concrete with 0.4% eqv. Na_2O cement may expand slowly if it contains small amounts of coarse opaline particles.
- Mortar-bar tests may be designed so as to be predictive for the behaviour of comparable concrete.

CONCLUSIONS

The more than a decade of early Australian CSIRO investments in ASR research is still a scholarly example of visionary, national willingness to sponsor and manage a long-term, holistic concrete research programme. The research leadership and the officers executed it with persistent dedication and determination to create an entity of knowledge which nowhere abroad became available while the programme was carried out.

Even today, after universal appreciation of ASR as globally occurring, the achievements of the programme ought to be earmarked in the ASR history as a systematic base of expertise, which has not been surpassed and made obsolete by later ASR research anywhere in the world.

The most unusual, meritorious feature of the research is the persistence from the commencement that the chemistry and mechanism of ASR are complementary aspects of the fundamental nature of the reaction and consistently must be investigated with this in mind.

The choice of mortar bar expansion test with opal as the reactive "model" aggregate material related the programme to common types of concrete aggregates in Australia and made it possible to investigate systematically the influence of the relevant parameters on the chemistry and mechanism of ASR. The researchers were aware, and made it clear, that other reactive rock types would require recognition of the different characteristics of such materials.

The knowledge that only special, unfavourable conditions engender harmful, i.e. expansive ASR, has a high mark as an early, pioneering achievement of the research. It implied correspondingly that harmless ASR must be expected to be the most common modification of the reaction in general concrete construction practice. Reasons therefore are deducible from the research observations and analyses.

The principal difference between the results of laboratory mortar expansion tests and the effects of ASR in field concrete were approached late in the programme. Comparable field investigations were apparently not possible, but reasonable assumptions for transfer of the achieved "model" knowledge was presented as guidelines for continued research elsewhere.

The basic kinetics and thermodynamics of the chemical reaction was not approached in the programme, although the acknowledged acceleration of the reaction at elevated temperature, and the implied conversion of chemical energy to the work released by swelling and crack formation in reacting aggregate particles indicated rewards by application of the basic scientific disciplines. The experienced termination of ASR in mortar bars was, incidentally, also an indication that the energetics of ASR might be a rewarding realm of further research within the holistic concept of the reaction.

In conclusion it deserves to be said that an effective publishing phase with emphasis on reader-attraction overseas in the wake of the termination of the work in 1958 would have been beneficial for much later ASR research in many countries during the ICAAR period. H.E. Vivian's reviews at some of the conferences, as referred to above, were always appreciated, but the conditions prevented presentations of the back-up documentation. In any case, the CSIRO concrete research was a great feat. The indefatigable execution of the individual research projects within a convincing overall architecture of the entity of issues was a second to none complex of integrated efforts.

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