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Title	Domain size as a parameter for studying the potential alkali-silica reactivity of chert-bearing aggregates
Authors(s)	McNally, Ciaran, Richardson, Mark G., Carr, A. J. (Alun J.), Strogon, Peter
Publication date	2004-05
Publication information	McNally, Ciaran, Mark G. Richardson, A. J. (Alun J.) Carr, and Peter Strogon. "Domain Size as a Parameter for Studying the Potential Alkali-Silica Reactivity of Chert-Bearing Aggregates" 56, no. 4 (May, 2004).
Publisher	Institution of Civil Engineers
Item record/more information	http://hdl.handle.net/10197/2434
Publisher's version (DOI)	10.1680/macr.2004.56.4.201

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Domain size as a parameter for studying the potential alkali-silica reactivity of chert-bearing aggregates

C. McNally,^{*} M. G. Richardson,[†] A. J. Carr[‡] and P. Stroger[§]

^{*} Materials Ireland, Department of Civil Engineering, University College Dublin, Republic of Ireland.

[†] Department of Civil Engineering, University College Dublin, Republic of Ireland.

[‡] Department of Mechanical Engineering, University College Dublin, Republic of Ireland.

[§] Department of Geology, University College Dublin, Republic of Ireland.

The presence of chert or flint in aggregates used for concrete manufacture has long been a source of concern in the context of alkali-silica reactivity. The global in-service performance of chert and flint-bearing aggregates, however, varies from innocuous to deleterious. This could be due to variations in the degree of crystallinity of the silica which influences potential reactivity. Chert occurs in significant Irish sources of aggregate used for concrete, yet no cases of deleterious expansion have been reported in the Republic of Ireland to date. Many of these sources fail to conclusively demonstrate innocuous behaviour in the laboratory expansion tests despite satisfactory in-service behaviour. A previous X-ray diffraction study, employing quartz crystallinity index, had indicated a significant difference between Irish Carboniferous cherts and English Cretaceous flints. This paper reports a further study of crystallinity, correlated with mortar bar expansion tests, but employing both quartz crystallinity index and domain size in the characterisation of crystallinity. The influence of chert content was also studied. Twenty-three Irish aggregate sources were sampled and petrographic analysis revealed that 17 of these contained chert. Three flint-bearing aggregates, from sources in England that had demonstrated alkali-silica reactivity, were also sampled. Chert and flint was extracted from the aggregate samples by a petrographer experienced in the field of concrete technology. Twenty-six distinct samples were successfully extracted from 13 of the Irish and the 3 English sources. X-ray diffraction testing confirmed the previous findings, (using the quartz crystallinity indices), that the Irish cherts are more crystalline than the English flints. Domain size determination however showed that the difference in crystallinity of the national sets was less than that anticipated. The influence of chert content is advanced as a hypothesis to explain the apparent reactivity of Irish cherts in screening tests. The potential value of combining domain size and chert content determination, in helping to classify aggregate reactivity, is advanced for cases where in-service behaviour is in significant conflict with findings from standard laboratory tests.

Notation

t	mean crystallite dimension - the domain size
K	Scherrer's constant
λ	wavelength of the x-rays
β	peak broadening at half maximum intensity due to low domain size
θ	diffraction angle of the peak in question
μ	mean value
Γ	full width at half maximum intensity
B	peak width at half maximum intensity of the diffraction pattern in question
b	peak width at half maximum intensity of the diffraction pattern of pure synthetic quartz

Introduction

Alkali-silica reaction (ASR) has been identified worldwide as a threat to concrete durability yet, despite international awareness of the reaction, it has continued to appear in countries previously unaffected by ASR. Microcrystalline quartz in the form of chert or flint is one of the principal causes of ASR. Despite this, no case of deleterious ASR has been identified to date in the Republic of Ireland, where many aggregates contain significant amounts of chert of Lower Carboniferous age and the former use of cements of relatively high alkali content. This is in contrast to the behaviour of some flints in similar climatic conditions in England, some of which reacted, with resultant expansions of a deleterious nature. A previous study by Stroger¹ demonstrated that the differing reactivity of Irish cherts and English flints could be explained by their distinct geological history. Independent geological estimates of the maximum temperatures reached by the host rocks to the cherts indicate that they have been subjected to temperatures ranging from just over 200°C to 350°C in different parts of the country. Quartz crystallinity correlates with the temperature reached by the host rocks, leading to the hypothesis that post-Carboniferous heating has annealed these cherts, making them relatively unreactive.

Alkali-silica reactivity is intrinsically linked to crystal structure. The degree of crystallinity varies in the forms of silica found in aggregates for concrete. Morino² reported that there is a relationship between the degree of crystal ordering and the potential reactivity of silica. The less ordered the crystal structure, the more reactive the silica. For example, Stanton *et al.*³ observed that opal can cause a damaging alkali silica reaction at levels as low as 1% due to the amorphous structure of opaline silica. Dent Glasser and Kataoka⁴ demonstrated that the disordered nature of the opal allows the hydroxyl ions to attack the silica at multiple locations, leaving it very prone to expansive reactions. Opal is almost completely amorphous, displaying no crystal structure whatsoever. Conversely quartz, with its hexagonal crystal structure, is quite stable in the presence of alkalis despite being entirely composed of silica⁵.

The ideal crystal shape of silica is a three-dimensional lattice of SiO₄ tetrahedra, in which each tetrahedron's oxygen is shared by adjoining tetrahedra as illustrated in Figure 1. However this is generally not the case, and the SiO₄ tetrahedra will take up different arrangements, leading to various crystal structures. Some varieties of silica that are relevant to alkali-silica reaction studies are quartz, tridymite, cristobalite, chert, chalcedony and opal. Chalcedony is a cryptocrystalline variety of silica, consisting of very fine crystals, and it is essentially fibrous in nature. Chert is also classified as cryptocrystalline silica, though the size of the crystals can increase as diagenesis proceeds.

Any crystal is composed of many individual domains, within each of which the crystal structure is perfect. Each domain however does not fit exactly with its neighbours but joins them through a variety of crystal defects. Figure 2 illustrates the role of these domains in forming a chert grain. If a chert crystal has small domain size, then many such defects will be present, most critically on the crystal surfaces where they form potential reaction sites. It seems reasonable to infer that smaller domain size will lead to more reactive cherts.

Domain size must not be confused with crystal size. Individual pieces of flint or chert are composed of multitudes of microcrystals of quartz, ranging from less than 2 microns to 30 microns in size in different cherts. Domains make up these microcrystals and are themselves two orders of magnitude smaller. Domain size and crystal size are not related.

Although finer crystal size ought, by the same logic, lead to increased reactivity, it has little effect. Petrographic examination of expanded mortar bars showed no correlation between crystal size and reactivity. Domain size seems to be the overriding factor in reactivity.

Strogen¹ has proposed that the reason for the absence of a case of ASR to date in Ireland is due to the Irish cherts being kinetically more stable than their U.K. flint counterparts. The reactive U.K. flints are Cretaceous in origin and have been exposed to temperatures of up to 200°C. In comparison Irish Carboniferous cherts have been subjected to temperatures from over 200°C to 350°C. The effect of the increased age and temperature is that the Irish cherts have been annealed, producing cherts that are significantly better than the reactive U.K. flints. It is postulated that it is from this enhanced crystallinity that the alkali resistance of Irish cherts is derived.

Sample Preparation

A selection of 23 aggregate sources representing a significant proportion of the concrete produced in Ireland was selected. Potentially reactive components were sought in each sample, with chert or flint identified as the primary potentially reactive materials of interest. Seventeen were found to contain chert and of these 13 yielded chert for the XRD analysis programme. Some of the aggregate samples displayed more than one variety of chert and these were separated and tested individually. Three potentially reactive aggregate sources from England were also introduced to the study for comparative purposes. The chert or flint content was determined for the 16 aggregate samples. The aggregates were subjected to petrographic analysis and the results are presented in Table 1. The Irish aggregates, prefixed by L and H, are listed in order of increasing chert content. The English flint-bearing aggregates are identified as samples PR-1, PR-2 and PR-3. Of the Irish aggregate samples selected, chert was identified in varying amounts. The chert content ranged from 1% to 30%. Some of the aggregates contained chert from a number of different sources. The English aggregates contained flint from various origins, with the content ranging 10% to 35%. Other potentially reactive components were identified, including greywacke and metaquartzite. Some of the aggregates also contained shale in varying amounts.

The quantity of chert required to run an X-ray scan is quite small but for the scans to provide meaningful results the chert must be extremely fine. Furthermore, to allow comparison between the results the chert samples needed to be of equal fineness. To achieve this the chert samples were crushed in a Fritsch Pulverisette, a small closed container with a steel ball, mounted on a table which causes the steel ball to vibrate, in turn crushing the chert. Following crushing, the chert was passed through a 45 µm sieve and the <45 µm material was used for scanning. Any chert remaining in the 45 µm sieve was further ground using an agate mortar and pestle until it all passed the sieve.

The diffraction profile peaks are fully developed only in diffractograms of well-crystallised quartz (Murata and Norman⁶). The peaks however will be distorted in the presence of other materials and to avoid this only samples of pure chert and flint were scanned.

For the purpose of analysing the results a standard quartz was tested as a reference. The sample chosen was Merck pure quartz *pro analysi* grade, (quartz fine granular, washed & calcined GR) and was prepared and scanned in the same manner as the above samples. As this is the standard by which the other samples would be judged, it was carried out in duplicate. This led to a total of 28 scans including a duplicate test on pure quartz, as set out in Table 2. Two distinct XRD methods were used to assess chert crystallinity - quartz crystallinity index (QCI) and determination of the domain size of the chert. QCI is determined according to the method of Murata and Norman⁶. The method for determination of domain size is described in the appendix to this paper.

X-Ray Diffraction Methods

The Guinier X-ray diffractometer used (Huber G642) was fully automated (using Huber G600 software) ensuring that all of the scans were performed in an identical manner. The only possible variation lay in placement of the chert sample in the diffractometer. The mounting that holds the sample is placed on a special holder and a small square of film is placed tightly across it. Some petroleum jelly is then smeared on the film and the crushed chert is then shaken onto the jelly and spread evenly. Care must be taken that a minimal amount of the jelly is placed on the film as an excessive amount will absorb some of the X-rays. It is worth noting that the petroleum jelly will also provide some X-ray diffraction peaks but that those peaks are located at a point on the spectrum far removed from the peaks belonging to the chert. For the sake of uniformity however, care was taken that the amount of petroleum jelly and crushed chert applied for each scan was as consistent as possible. The diffractometer features an oscillator that ensured that all parts of the chert sample were reached by the X-rays. The X-ray settings were the same for all of the scans.

For the purpose of these tests, the X-ray scans were carried out using the same intensity over a range of incident angles from 33.75° to 34.75°. The diffractometer was set to record the intensity of the diffracted beam, in counts per second, at intervals of 0.01° and with a count time of 2 seconds. The output from the scans came in the form of a diffraction pattern that the software accompanying the diffractometer wrote to a data file. From this diffraction pattern, it is possible through different procedures to determine the QCI and domain size for each chert sample.

For QCI determination the scans were smoothed (using the Savitsky-Golay method) and the height of the 212 peak over low and high angle background radiation was measured **using the method described in the appendix**. Examples of XRD scans for moderately crystalline and poorly crystalline material are shown in Figures 3 and 4 respectively. This procedure was followed for all chert and flint samples listed in Table 2. Pure quartz was included as the basis of the QCI is that a pure quartz sample will produce a value of 10. Unfactored QCI values for pure quartz were determined as 8.4 and 8.1 for Scans 27 and 28 respectively. These yield factors of 1.190 and 1.235 to bring the values to 10. The average factor, 1.213, was applied to the other

unfactored results to calculate the required index. The results from QCI determination are presented in Table 3.

From the pure quartz sample, analysed in duplicate, a value of b was determined. The results of the tests on quartz are presented in Table 4 from which an average full width at half maximum intensity (FWHM) for the 212 peak of 0.0575° , in terms of θ , which for use in the Scherrer equation was converted to 2θ and expressed in radians. For domain size determination B is measured as the FWHM for each diffraction profile and β is then calculated using Equation 4 **as described in the appendix**. The results of domain size determination are presented in Table 5.

Results and discussion

Consideration of varieties of chert and flint

In some of the aggregates studied, petrographic analysis showed the existence of a number of different cherts or flints. These sources were the high chert content Irish aggregates H-2, H-4 and H-6 and the English aggregates PR-1 and PR-3.

The fine fraction of aggregate H-2 was dominated by quartz and contained a black sedimentary chert and a white volcanic chert. Both cherts were extracted for QCI and domain size determination. They returned QCI values of 6.4 and 7.1 and domain sizes of 604 and 693 Å respectively. Petrographic analysis of the fine aggregate H-4 detected the presence of a grey-white chert and a black chert. These cherts returned QCI values of 6.4 and 6.3 and domain sizes of 525 and 499 Å respectively, indicating that again the different cherts are of a consistent crystallinity.

The coarse aggregate H-6 contained a number of different cherts and four samples were extracted for X-ray analysis. The QCI values for these cherts were 7.6, 8.0, 8.0 and 7.1 while the corresponding domain sizes were 656, 862, 885 and 614 Å. These are some of the highest values produced in this study and may indicate that the cherts present in aggregate H-6 were already very crystalline.

The fine aggregate sample PR-1 produced three distinct flint samples. These samples varied in colour and degree of weathering and petrographic analysis indicated that they came from a variety of different sources. The QCI values were 1.4, 2.1 and 3.4 while the corresponding domain sizes were 274, 321 and 382 Å. Considering these all occur in one aggregate, the range of QCI values is quite wide. The fine aggregate PR-3 returned four flint samples which ranged in their degree of weathering but the characteristics of the flints were very consistent, returning QCI values of 3.1, 1.8, 2.1 and 2.2 while the domain sizes were 319, 266, 271 and 276 Å. Thus the weathering effect in the English flints does not appear to be as significant as the thermal history of the Irish cherts.

Quartz crystallinity index and domain size

A strong relationship was apparent between QCI and domain size. In areas where the domain size was lowest, *circa* 275 Å, so too was the QCI value, *circa* 1.5. An approximately linear relationship was exhibited up to a domain size of about 600 Å, at which level the QCI was about 7.0. Above this value however the relationship loses its linearity. This is not unexpected since the domain size can continue to increase indefinitely whereas the QCI is limited to a maximum value of 10.0.

Analysis of the QCI values produced show that there is a difference between Irish cherts and English flints. The Irish cherts had QCI's between 5.9 and 8.1 with an average of 7.0. The reactive flints had an average QCI of 2.2, the values ranging between 1.4 and 3.1. Thomson and Grattan-Bellew⁷ state that the crystal structure and grain size/surface area contribute to the total free energy of the material. **In a perfectly crystalline lattice, the free surface energy is minimised, and any deviation from crystalline perfection increases the free surface energy of the material.** Smaller domain size means that the number of dislocations will be higher in the reactive flint than in the Carboniferous Irish chert. This leads to an increase in the internal surface area, leading to a notable difference between the free energy of the reactive flint and that of the Irish chert. **It is this increased free energy that makes the reactive flints unstable relative to the Irish cherts.**

The comparison of Irish and English samples bear out the statement by Dent Glasser and Kataoka⁴ that increased crystal perfection implies enhanced resistance to alkali-silica reaction. However the lowest domain sizes found in Irish aggregates was surprising. The Irish cherts possessed domain sizes ranging from a low of 487 Å to a maximum of 885 Å. The English flints produced lower domain sizes, ranging from 214 Å to 382 Å. The expectation from the earlier QCI study by Stroger¹ and the absence of reported cases of ASR in structures in service was that all Irish cherts would have a significantly increased domain size relative to the chosen reactive flints due to the extended thermal maturation processes. While the domain size of the Irish cherts was greater than that of the English flints, the difference was not pronounced at the lower and upper limits respectively. However, it must be borne in mind that the relationship between domain size and volume is cubic. The effect of doubling the average domain size is to reduce the number of domains in a given volume of crystal by a factor of 8. Although the study confirmed the earlier finding of a difference between the crystalline characteristics of Irish cherts and reactive English flints, the difference in crystallinity of some of the Irish cherts and English flints was not wide enough to provide a comforting margin. Other factors, such as chert content require consideration.

Effect of both crystallinity and chert content

The crystallinity of the chert or flint present in an aggregate sample will play a large role in its reactivity. However, the aggregate sample may not be expansive if the potentially reactive chert is not present in sufficient quantity. The chert may be reactive but the amount of gel produced, and the resultant expansion, may be inconsequential if the amount of reactive chert present is low. Damaging expansion due to ASR is dependent on the presence of a significant supply of reactive silica to sustain the reaction.

The RILEM ultra-accelerated mortar bar test⁸ has been developed as a screening test for alkali reactivity. The test may be used to give early warning of potential reactivity by individual aggregates, the true behaviour of which may be more fully examined by concrete prism tests. The aggregate samples that contained the chert and flint had also been tested individually using the RILEM ultra-accelerated mortar bar test (McNally and Richardson⁹). While some of the aggregates tested may have included other potentially reactive material it was concluded that the primary potentially reactive constituent was the chert and flint. It is interesting to review the results of these

studies with those of the domain size study together with the petrographic assessment of chert content. The results are presented in Table 6.

Despite the small sample size some correlation was apparent between the domain size of the chert, the chert content and the expansion category. For example large domain sizes combined with moderate chert content (H-3) led to low expansion levels, whereas low domain size combined with high chert content (PR-1) led to high expansion levels. Some aggregates contained relatively low domain size chert with low chert content, for example L-8, and petrographic thin sections (such as that in Figure 5) showed that while reaction had occurred the expansion level was not high. It was also apparent that domain size or chert content alone is not sufficient to predict the likelihood of damaging expansion.

It would appear that for a chert-bearing aggregate to suffer damaging alkali reactivity there are two conditions required:

- the chert present must have low domain size to allow alkali attack;
- the chert must occur in sufficient quantities so as to produce expansion.

Thus determination of domain size may be of value in the assessment of aggregates that yield borderline results in standard tests. An initial simplified structure for risk assessment is presented in Figure 6, which illustrates boundaries based on a small sample size.

Conclusions

The study confirms that determination of crystallinity may be helpful in differentiating the likely reactivity of chert-bearing aggregates. Quartz Crystallinity Index is capable of detecting the large-scale difference that occurs between well crystalline and poorly crystalline silica but is not precise enough to differentiate cherts of intermediate crystallinity. However domain size is a suitable parameter for differentiating cherts of similar order of crystallinity. For a given chert content it would appear that domain size may be a valuable parameter in making a judgement on alkali reactivity of chert-bearing aggregates where results from standard tests are borderline.

Appendix - Quartz crystallinity index and domain size determination

Murata & Norman⁶ developed the Quartz Crystallinity Index (QCI) as a straightforward tool for comparative studies of cherts. The basis for the tool is the fact that in the X-ray diffraction (XRD) profile for quartz, the 212 peak is poorly developed in quartz of lesser crystallinity. The QCI is calculated from the ratio of the relative height of the 212 peak over the high angle background to that over the low angle background (Figure 3). A perfectly crystalline quartz sample is taken to have a QCI of 10. To normalise the QCI, all of the results are multiplied by a factor determined from the diffraction pattern of an ideal quartz sample. To obtain this information, XRD scans are carried out around the 212 peak, at a d-spacing of approximately 1.376 Å. The scans are complicated however by the presence of the 203 and 301 peaks with d-spacings of approximately 1.369 Å and 1.366 Å respectively.

Klug and Alexander¹⁰ and Cullity¹¹ state that in X-ray diffractometry small domain size leads to broadening of the X-ray diffraction profile. By using the diffraction profile of a perfectly crystalline quartz sample in conjunction with the diffraction profile, it is possible to determine the domain size of the chert sample. The diffraction pattern is broken down into three distinct peaks, and these peaks are used to determine the domain size of the chert or flint sample being analysed.

The peaks can be modelled as a series of mathematical distributions. Using the distributions extracted from the diffraction profiles, it may be possible to determine the domain size by use of the fact that a decrease in domain size will be indicated by a broadening of the distributions. Pure synthetic quartz, which has a very large domain size due to its highly crystalline structure, produces a diffraction pattern of high, sharp peaks. The diffraction pattern of a less crystalline material exhibit this loss in crystallinity by the peaks becoming lower and broader.

Klug and Alexander¹⁰ have described how, if a crystal is assumed to have no lattice imperfections, any broadening of the pure diffraction profile can be attributed solely to small crystallite size, and theoretical equations may be used to determine the mean crystallite dimension. The Scherrer equation may be used. The Scherrer equation for this is:

$$t = \frac{K \lambda}{\beta \cos \theta}$$

Equation 1

The broadening effect due to small domain size (β) is calculated from the change in the diffraction profile relative to the pure diffraction profile. It is expressed in terms of 2θ in radians.

The diffraction profile is deconvoluted by modelling the three peaks as mathematical distributions. The peaks may be modelled as either a Gaussian or Lorentzian distribution. The actual peak profile will lie somewhere between the two¹⁰. In practice, the data profile was best modelled as a series of Lorentzian distributions (Figure 3). The encircled points correspond to the counts recorded by the

diffractometer and represent the raw data. The QCI can be calculated from the raw data but more information is required to calculate the domain size of the sample.

The three narrow line profiles represent the Lorentzian distributions, which when summed approximate a profile of the raw data. The heavy line profile represents the sum of the three Lorentzians. The dashed line profile is a graph of the residual difference between the raw data and the mathematical estimate. It can be seen that this residual moves above and below a straight line, indicating a positive and negative difference between the raw data and the mathematical model.

The Lorentzian distribution is represented by Equation 2.

$$P(x) = \frac{1}{\pi} \frac{1/2\Gamma}{(x - \mu)^2 + (1/2\Gamma)^2}$$

Equation 2

In relation to Equation 1, the wavelength of the X-rays (λ) is 1.54056 Å. The broadening effect (β), which may be determined either by Equation 3 of Klug and Alexander¹⁰ or by Equation 4 of Cullity¹¹:

$$\beta = B - b$$

Equation 3

$$\beta^2 = B^2 - b^2$$

Equation 4

Equation 3 is based on a Gaussian distribution while Equation 4 assumes the profile to be Lorentzian. The two approaches are almost identical in determining the mean crystallite size, the only difference being in the method of calculating the broadening effect.

The peaks are thus modelled as a series of distributions and it is possible to determine the domain size, using the methods of Klug and Alexander and of Cullity, using each of the three peaks. However the 203 peak and the 301 peak lie very close together. It is possible that this proximity may cause the two peaks to interfere with each other, and thus distort the results. There is a high probability that this proximity will lead to interference when the doublet is resolved into a pair of distinct peaks. When the computer software determines each curve, it does so while reducing the overall error to a minimum. In resolving two peaks that lie very closely together, it is possible that the resolution of one peak will be to the detriment of the other. Due to this, domain size was based on the broadening of the 212 peak alone, the peak most affected by domain size.

The Lorentzian distribution provided a better fit to the data produced and so Equation 4 was used to calculate the broadening effect (β). Using the diffraction profile for pure quartz and resolving all three peaks in all scans, it is possible to determine the domain size for all chert samples.

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