# Quantitative Evaluation of Rejuvenators to Restore Embrittlement Temperatures in Oxidized Asphalt Mixtures using Acoustic Emission

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## Abstract

Towards developing a method capable to assess the efficiency of rejuvenators to restore embrittlement temperatures of oxidized asphalt binders towards their original, i.e., unaged values, three gyratory compacted specimens were manufactured with mixtures oven-aged for 36 hours at 135°C. In addition, one gyratory compacted specimen manufactured using a short-term oven-aged mixture for two hours at 155°C was used for control to simulate aging during plant production. Each of these four gyratory compacted specimens was then cut into two cylindrical specimen 5 cm thick for a total of six 36-hour oven-aged specimens and two short-term aging specimens. Two specimens aged for 36 hours and the two short-term specimens were tested using an acoustic emission approach to obtain base acoustic emission response of short-term and severely-aged specimens. The remaining four specimens oven-aged for 36 hours were then treated by spreading their top surface with rejuvenator in the amount of 10% of the binder by weight. These four specimens were then tested using the same acoustic emission approach after two, four, six, and eight weeks of dwell time. It was observed that the embrittlement temperatures of the short-term aged and severely oven-aged specimens were -25°C and -15°C, respectively. It was also observed that after four weeks of dwell time, the rejuvenator-treated samples had recuperated the original embrittlement temperatures. In addition, it was also observed that the rejuvenator kept acting upon the binder after four weeks of dwell time; at eight weeks of dwell time, the specimens had an embrittlement temperature about one grade cooler than the embrittlement temperature corresponding to the short-term aged specimen.

**Keywords:** Asphalt concrete, oven-aging, embrittlement temperatures, asphalt binder, rejuvenator

## 1. Introduction

In the United States, there are ongoing efforts to develop more efficient maintenance strategies for asphalt pavements to help achieve both economic and environmental goals. It has been estimated that about 96% of the approximately 4 million km of paved roads in the country are surfaced with asphalt (Roberts, 2002). The popularity of asphalt concrete derives from the fact that it delivers a smooth, quiet surface, and can be rapidly constructed, particularly during rehabilitation, i.e., resurfacing, operations. Immediately after construction, asphalt is a remarkably tough and resilient material. This is mainly due to the fact that asphalt concrete is comprised of a highly ductile and healable matrix, i.e. asphalt binder, combined with hard aggregate particles, i.e., crushed stone, which provides stiffness and strength to the system. When compared to Portland cement concrete, asphalt concrete can possess 5, 10, or even 20 times more fracture energy due to its ductile binder matrix and toughness-adding aggregate structure. Although traffic loads and thermal cycles tend to cause micro-damage in the asphalt binder system under certain conditions, this damage can be healed provided the binder retains sufficient fluidity. As a result, even as the pavement develops minor rutting and distributed micro-damage, pavement serviceability and smoothness can remain at a very high level for many years of service.

However, with time, asphalt binder ages, particularly near the pavement surface, which causes the binder to lose its ductility and resilience. Oxidative hardening leads to stiffness and embrittlement of asphalt binders, see Fig. 1, which reduces healing capacity, and increases the rate of micro-crack propagation. Furthermore, the pavement system is more prone to coalesced micro-crack formation, and may begin to develop surface-initiated fatigue cracking. In addition, the brittle pavement surface will be prone to channeling cracks, such as thermal and block cracks, see Fig. 2. Fatigue, thermal, and block cracking lead to an exponential decline in pavement serviceability and a resulting exponential increase in maintenance costs to restore pavement condition. Furthermore, in a recent study conducted by Islam and Buttlar (2012), a rough pavement network was found to add an additional user cost of over $300 per vehicle per 19,000 km driven. The encouraging news from the study was that properly timed maintenance treatments, resulting in moderately smooth pavement over its life, yield approximately a 50-to-1 return on investment. Life extension has also been linked to sustainability benefits (reduced energy and emissions in the life cycle of the pavement from cradle to grave).

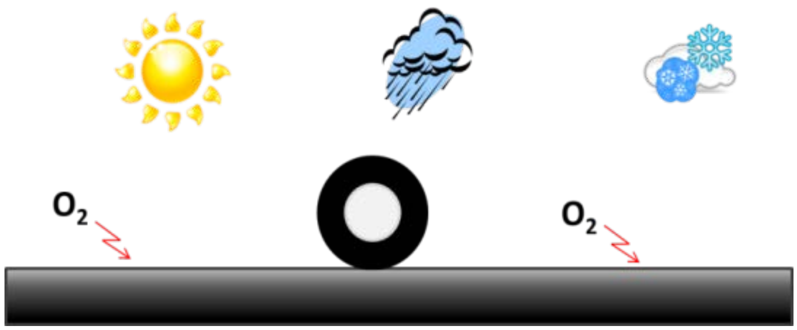


Fig. 1. Oxidative aging of asphalt concrete pavements. With increasing oxidative aging in the top material layer, the pavement becomes increasingly more susceptible to damage accumulation due to mechanical and environmental loads.

Fig. 2. Thermal cracking (left) and block cracking (right) on asphalt concrete pavements in Illinois.

*In-situ* field aging or long-term aging is the dominant source of material property gradation through the pavement thickness. Figure 3 shows the predictions made using a binder aging prediction model by Mirza and Witczak (1995) for an eight-years old asphalt pavement subjected to central Illinois climactic conditions. Figure 3 illustrates the highly graded material properties caused by oxidative hardening through the pavement thickness. The time required to reach an unacceptable level of embrittlement near the pavement surface depends upon a number of factors, and varies from pavement to pavement, even within a given region and mixture type. Reliable tools are therefore needed to characterize the steeply graded properties of an aged asphalt pavement (Dave, 2009) and intelligently select the best maintenance strategy to restore the pavement to a crack-resistant state, which may include optimizing the relative amount of milling and surface replacement and/or the use of rejuvenators.

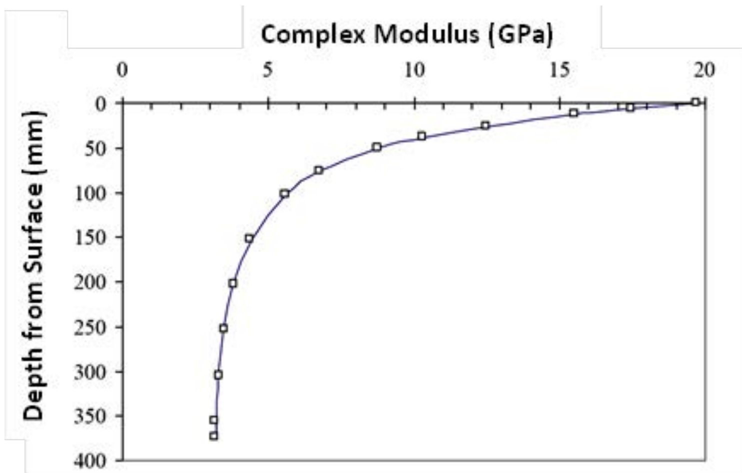


Fig. 3. Asphalt concrete complex modulus after eight-years in service, as predicted using Mirza and Witczak's global aging model (Mirza and Witczak, 1995).

The use of rejuvenators is currently becoming popular. Their function is to restore the properties of stiff, highly oxidized (i.e., aged) bitumen by softening the aged asphalt, for instance, via the restoration of the original asphaltenes-to-maltenes ratio. In this study, an acoustic emission based approach is used to quantitatively evaluate the efficiency of rejuvenators in restoring the embrittlement temperature of the oxidized mixtures to its original values.

*1.1. Rejuvenators: A Literature Review*

The hard, oxidized nature of reclaimed asphalt binder is a major concern when incorporating recycled materials into HMA mixtures. Oxidation—the main mechanism behind asphalt aging—changes certain chemical properties of the asphalt; specifically, the ratio between asphaltenes and maltenes. Asphaltenes are defined by their insolubility in pentane and function as bodying agents, whereas maltenes are the residual constituents after asphaltene precipitation (Long, 1982; Boyer, 2000; Buenrostro, 2001). Types of maltenes include polar compounds/nitrogen bases (peptizers for the asphaltenes), acidiffins (solvent for the peptized asphaltenes), and paraffins (saturated hydrocarbons that act as a gelling agent). The asphaltenes to maltenes ratio influences the rheology of bitumen; as oxidation occurs, polar compounds and acidiffins are converted into asphaltenes thus increasing the ratio, resulting in a stiffer, more brittle asphalt binder. The reaction rate of oxidation can be accelerated at high temperatures and/or high exposure to ultraviolet light. The amount of oxidation is also positively correlated to how much asphalt is exposed to air; therefore, stockpiling recycled asphalt materials may result in more oxidation (Karlson, 2006). Regardless of the reasons for oxidation, products have been produced to counteract the effects of oxidation. Depending on the use of the product (preventative/corrective maintenance or recycling) these products have been called many different names (Karlson, 2006), such as "service life extenders", "softening agents," "rejuvenator seals," and "recycling agents/additives". For consistency, any such product will typically be referred to as a rejuvenator.

A rejuvenator, as the name implies, is a product that aims to restore the physical and chemical properties of aged bitumen. Rejuvenators address the issue of oxidative hardening by softening the aged asphalt via the restoration of the original asphaltenes to maltenes ratio discussed above (Buenrostro, 2001; Shen, 2007; Garcia, 2010). Some examples of rejuvenators are refined tallow, waste vegetable or frying oils, waste motor oils, lube extracts, extender oils, emulsions, soft virgin binders, and bio-binders (Buenrostro, 2001; Nahar, 2014; Oldham, 2014). Rejuvenators are generally applied to the surface of existing pavements; therefore, it is essential for the rejuvenator to have the ability to penetrate the surface and diffuse through the aged asphalt. If the rejuvenator lacks this ability, not only will the aged asphalt be unaffected, but the unabsorbed rejuvenator will reduce skid resistance (Garcia, 2010; Brown, 1988). To avoid creating slick, over-coated surfaces, it is often good practice to apply rejuvenators in several coats at a lower application rate (Boyer, 2000). During the diffusion process, the rejuvenator first forms a low-viscosity layer around the layer of aged binder which coats the aggregate. Then, the rejuvenator starts to diffuse into the aged binder, thus softening it. Eventually, all the rejuvenator penetrates into the aged binder and the inner layer becomes less viscous and the outer layer becomes more viscous as the mixture approaches a state of equilibrium (Garcia, 2010; Brown, 1988). Oliver (1974) found that the rate of diffusion can be increased by adding diluents or by increasing temperature. Thus, the environment in which the rejuvenator is applied is a critical consideration, especially in terms of application rate. After a sufficient dwell time, the performance of the rejuvenator can be evaluated.

Rejuvenators are also often used in conjunction with hot in-place recycling (HIPR) techniques. The goal of hot in-place recycling is to correct surface distresses in surface layers of HMA pavements while significantly reducing the need to haul new materials. During HIPR, the surface layer of the existing HMA pavement is heated, scarified, and then mixed with rejuvenators and/or softer virgin binder. This recycled material is then used to repave the roadway from which it was removed. The addition of rejuvenators aims to soften the aged asphalt and thus improve future cracking performance (Oliver, 1974). An example of the equipment used to perform these tasks is the MARTEC Recycling Corporation's forced hot air heating system (Crawley, 1999), which was first used in the United States in Mississippi on an interstate highway project from December 1997 to July 1998. Refer to Shoeberger *et al.* and Button *et al.* for more detailed overviews of HIPR procedures (Shoenberger, 1990; Button, 1999).

Research efforts are still being carried out towards developing a quantitative understanding of the effect of rejuvenators (Rostler, 1970; Scofield, 1986; Shen, 2006; Lin, 2012; Elseifi, 2012). The reader is encouraged to review these studies regarding the use of rejuvenators including the use of rejuvenators in conjunction of recycled asphalt pavement (RAP) (Shen, 2007) and recycled asphalt shingles (RAS) (Elseifi, 2012). There are many different types of rejuvenating products available, and some fulfill their function of softening hard asphalt better than others. Regardless, it is clear that the intended purpose of these products make them very desirable to be used in tandem with recycled materials (Beach, 2013; Tran, 2012; ASTM D7313-13). Based upon these studies, the effectiveness of rejuvenators is still an active area of research.

## 2. Experimental Description

An AE-based approach (Behnia, 2014; Buttlar, 2011; Dave, 2011; McGovern, 2013a, 2013b; Apeagyei, 2009) to estimate the embrittlement temperatures of oven-aged asphalt concrete specimens treated with rejuvenator is used. The AE-based approach to estimate embrittlement temperatures has the potential to replace the ASSHTO standard protocols (ASSHTO MP1, 1998;

ASSHTO TP1, 1999; ASSHTO MP1A, 2001) used to specify performance grading of asphalt binders and asphalt concrete mixtures at low-temperature because of its many advantages. A comparison between the AE-based embrittlement temperatures with those quantitatively evaluated using the ASSSHTO protocols can be found in (Apeagyei et al, 2009; Behnia, 2014; Buttlar et al, 2011; McGovern et al, 2013). The main advantages of the AE-based approach include the potential for portable instrumentation and rapid field testing. The embrittlement temperature of a binder or mixture is quantitatively evaluated by observing the AE event and AE event rate response caused by the increasing thermal stresses, which develop as the specimen cools because of the different coefficients of thermal expansion between the aggregates and the binder. When the magnitude of the tensile stresses reach the local binder strength, cracks occur, releasing strain energy in the form of transient stress waves, i.e., acoustic emission events, which are detected using piezoelectric sensors. Here, the temperature at which the event-rate is equal or above 5 events per second (with event energy equal or above 4 $V^2$-μs) is termed the mixture's embrittlement temperature. Figure 4 shows a schematic diagram of the two main types of thermally-induced microdamage in asphalt concrete, i.e., microcracks in the mastic and mastic aggregate debonding where mastic is the mixture of asphalt binder and fines. To visualize this type of micro-damage, x-ray computer micro-tomography imaging was taken of asphalt concrete specimens before and after cooling. Figure 5 shows the results of the micro-tomography imaging where mastic micro-cracking and mastic-aggregate debonding are observed. The length of the observed microcracks and debonding varied between 28 and 750 μm.

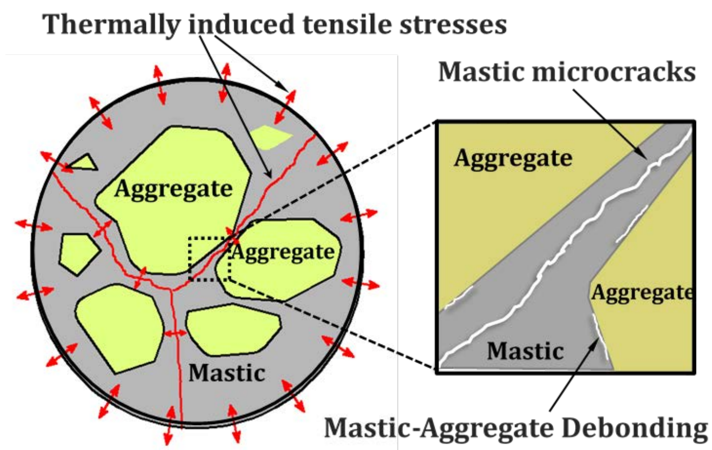


Fig. 4. Schematic illustration of two main types of microdamage within asphalt concrete, i.e., mastic microcrack and mastic-aggregate debonding.

*2.1 Preparation of test specimens*

Four different asphalt concrete samples were prepared using the same mix design. The mix was composed of 19 mm nominal maximum aggregate size (NMAS) and with a target asphalt content of 5.9% by weight of the total mixture. The binder used was Superpave PG 64-22 binder, where PG is an acronym for performance grade. The aggregate blend consisted of aggregates from four different stockpiles commonly used in Illinois: coarse (CM16), fine (FM20, FM02), and mineral filler (MF). Note that the mixtures conformed to Superpave mix design requirements. The mixing and compaction temperature (155ºC) was chosen to ensure proper fluidity of the binder. After batching and mixing heated aggregates with heated asphalt, the sample was spread evenly in a stainless steel pan and placed in the oven for 2 hours at 155 °C to simulate short-term aging according to AASHTO PP2 protocol. Short-term aging simulates the aging, which occurs during plant production of the mixture during construction.

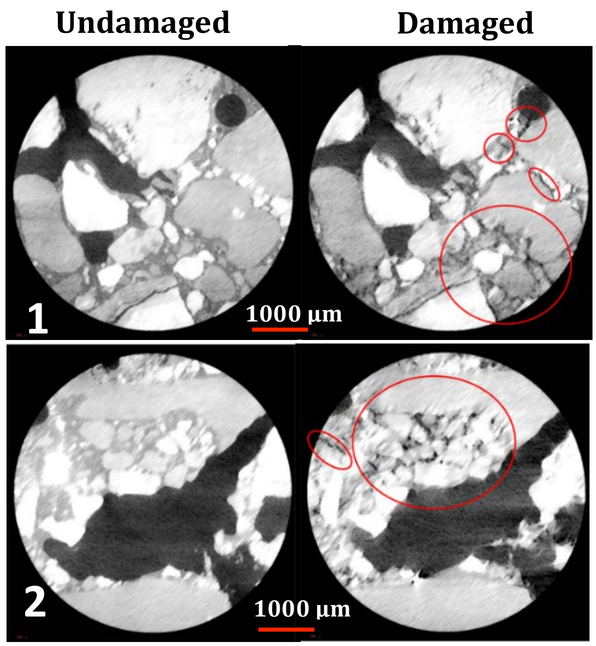

Fig. 5. X-ray computed micro-tomography imaging of crack damage induced by cooling. On the left are the images before cooling and on the right are the images after cooling. Both photographs show microcracks in the mastic microcrack and mastic-aggregate debonding.

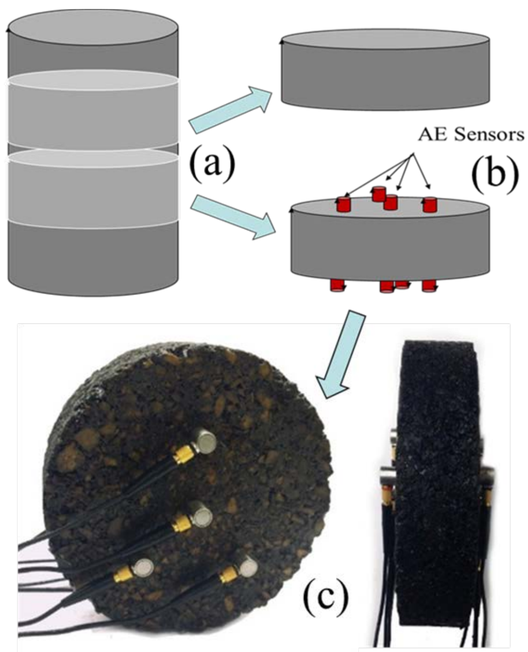

Fig. 6. Specimen preparation; (a) one gyratory compacted specimen of the left, (b) two 5-cm thick cylindrical specimens for AE testing on the right -- the lower specimen on the right also shows the four AE sensors on each side of the specimen, and (c) front and side photograph of a specimen, two weeks after being treated with rejuvenator, and showing four AE sensors coupled to each of the two flat surfaces.

Specimens were then subjected to long-term oven-aging for 36 hours by moving the pans of material into an oven set to 135ºC. To ensure uniform aging, the mixtures were hand-stirred every 12 hours. Four cylindrical samples were prepared using 4.5 kg of the aged mix. In addition to the long-term aged specimens, a control mixture was made using short-term aged material.

A servo-controlled Superpave gyratory compactor (IPC Servopac) was used for compaction of cylindrical specimens to a prescribed level of air voids. The samples were compacted using at least 100 gyrations to a height of 150 mm. Once the gyrations were complete, the mold was removed from the compacter. The specimens were then removed from the mold and allowed to cool to room temperature.

Once compacted and cooled to room temperature, the four gyratory compacted cylindrical specimens were cut to obtain eight, 5-cm thick cylindrical specimens with a diameter of 15 cm; six of these specimens were made with mixture that was oven-aged for 36 hours and two were made with a short-term aged mixture. Figure 6 shows the specimen geometry. During cutting, the ends of the gyratory compacted specimens were trimmed off in order to obtain a smooth surface for sensor placement.

Figure 6a illustrates how two, 5-cm thick cylindrical specimens were cut from one gyratory compacted specimen. Figure 6b shows how the eight AE sensors were coupled to the specimen, i.e., four on each side of the specimen. Two long-term aged specimens and two short-term aged specimens were used for control and tested using the AE-based approach. The remaining four specimens made with long-term oven-aged mixtures were treated on the top surface with rejuvenator by spreading rejuvenator on the surface in the amount of 10% of the binder content by weight. These specimens were then tested using the AE-based approach after the dwell time of two, four, six, and eight weeks. The short-term aged specimens and the specimens oven-aged for 36 hours were also tested using the same acoustic emission procedure for comparison and for calibration of the AE-based approach. The rejuvenator (donated by The Heritage Group) used was an emulsion of petroleum oils and resin called Reclamite®. Figure 6c shows a photograph of a specimen two weeks after being treated with rejuvenator. Figure 6c also shows four acoustic emission sensors coupled to each of the two flat surfaces of the specimen.

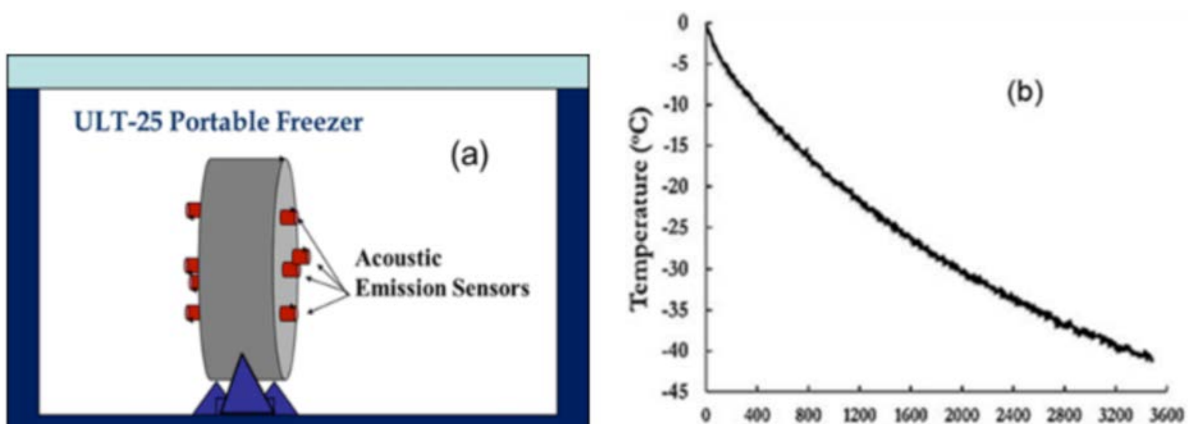


Fig. 7. Acoustic Emission testing during cooling: (a) Schematic diagram of the cooling chamber (ULP-25 Portable Freezer) and test specimen showing four AE sensors coupled to each of the two flat surfaces and specimen support (specimen and cooling chamber not to scale), and (b) typical temperature versus time curve.

*2.2 Acoustic emission (AE) testing*

The asphalt concrete embrittlement temperatures of the test samples were quantitatively evaluated by recording the AE test samples' AE response to thermal cooling from 15°C to –50°C, see Fig. 7. Figure 7a shows the schematic diagram of the cooling chamber (ULP-25 Portable Freezer) with one specimen with four AE sensors coupled to each of the specimen's two flat surfaces. The specimen was placed on felt pads to minimize vibrations from the cooling chamber and to allow cool air to circulate under the specimen to increase the cooling rate. The temperature was monitored and recorded using a K-type thermocouple, which was connected to one of the parametric inputs channels on the AE system unit via a K-type thermocouple adapter. The thermocouple was coupled directly to the surface of the test specimen using high vacuum grease. Figure 7b shows a typical sample cooling curve for the AE specimen.

Eight wideband AE sensors (Digital Wave, Model B1025) with a nominal frequency range of 50 kHz to 1.5 MHz were coupled directly to the surface of the test specimen using high-vacuum grease; four AE sensors were coupled on the top surface, i.e., on the surface treated with rejuvenator, and four AE sensors were coupled on the bottom surface, i.e., on the surface that was not treated with rejuvenator, see Fig. 6. Prior to testing, the AE sensors were conditioned in the cooling chamber to eliminate the AE events that arise due to the different rates of thermal expansion of the sensors' materials by keeping the sensors at -50$^{o}$C for the time period of two hours. The signals from the AE sensors were pre-amplified by 20 dB using a broadband pre-amplifier. The signals were then further amplified 21 dB (for a total of 41 dB) and filtered using a 20 kHz high-pass double-pole filter. A pre-trigger was used, and the waveforms were digitized using a 16-bit analogue-to-digital converter (ICS 645B-8) using a sampling frequency of 2 MHz. A total of up to 2,048 points were used to calculate the energy, provided the points were within the event duration, i.e., between the first and last overshoot crossings. The channels were triggered when the signal penetrated the threshold on one channel, and a threshold voltage of 0.1V was used. Figure 8 shows a typical AE time domain record of an event and the corresponding spectral content. To eliminate noise, all signals with an AE energy lower than 4 $V^2$-µs were filtered out. For additional information regarding AE-based estimation of embrittlement temperatures of HMA mixtures the readers are referred to references (Buttlar, 2011; Dave, 2011; McGovern, 2013a, 2013b; Apeagyei, 2009).

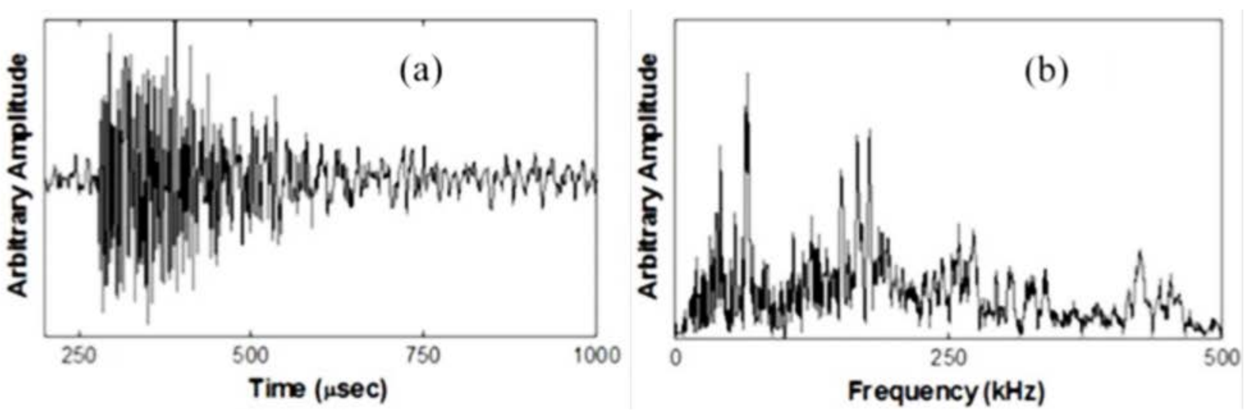

Fig. 8. Typical acoustic emission: (a) time domain record, and (b) corresponding spectral content.

The temperature corresponding to an event-rate equal or above five events per second (of events having an energy equal or above 4 $V^2$-µs) is termed the mixture's embrittlement temperature. Note that occasionally a test will produce a few isolated AE events above 4 $V^2$-µs early on in the test at warmer temperatures. Because these events are isolated, i.e., AE events do not occur at a regular rate after such an event as the specimen continues to cool—they are neglected and not considered to be the mixture embrittlement temperature.

## 3. Experimental Results and Discussion

The stochastic nature of asphalt concrete with two distinct phases, i.e., mastic and aggregates, where each phase has different acoustic properties, leads to energy scattering and mode conversion. This effect increases with frequency. The presence of multiple scatterers makes it possible to have multiple mode conversions and multiple paths of the (primary and scattered) wave fields from the source location to the observation point. As a result, both the bulk dilatational and shear velocities and the corresponding attenuations depend upon the frequency and the level of aging. McGovern et al. (2013a, 2013b) provide a very good discussion of the difficulties in estimating velocities and corresponding attenuations in asphalt concrete. Both the short-term aged specimens and the specimens oven-aged for 36 hours had uniform properties through their thickness. However, in the case of the oven-aged specimens for 36 hours exposed to rejuvenator on the top surface, the material properties of the specimens became graded through their thickness as the rejuvenator acted upon the aged binder throughout the thickness of the specimen. As a result, the longitudinal and shear velocities and the corresponding attenuations also became graded throughout the specimen's thickness.

For asphalt concrete mixtures without the presence of rejuvenator, the AE-based embrittlement temperature is determined as the temperature at which an event with an energy equal or above a prescribed value occurs. The prescribed energy value is determined using a mixture of known value for its embrittlement temperature. For these cases, the specimen volume is irrelevant, provided it is large enough to provide constraints to the binder contraction during thermal cooling. This occurs when the specimen size is about four-to-five times the size of the larger aggregate (Apeagyei et al, 2009; Buttlar et al, 2011; McGovern et al, 2013).

For asphalt concrete mixtures in the presence of rejuvenator, the specimens became graded structures whose gradation is a function of time. The penetration of the rejuvenator into the specimen depends upon the porosity distribution and the corresponding tortuosity, which is typically not uniform within the volume of the specimens. As a result, at a given time there are pockets of asphalt concrete within the test specimens where the rejuvenating effect did not yet occur. Therefore, these pockets of asphalt concrete have warmer embrittlement temperatures than the material that has already been "rejuvenated." During thermal cooling these pockets of warmer embrittlement temperatures produce sporadic energetic AE events, which makes the evaluation of the specimen material embrittlement temperature more difficult. To avoid this difficulty, i.e., the influence of these sporadic energetic events, a rate criterion was developed where the embrittlement temperature is defined as the temperature when a prescribed event rate occurs where the events have an energy equal of above a prescribed value. The prescribed event energy and the event rate need however to be calibrated, which in this study is done by testing two mixtures with known embrittlement temperatures, i.e., the short-term aged mixture and the oven-aged mixture for 36 hours, which have embrittlement temperatures of -25$^{o}$C and -15$^{o}$C, respectively. Because a larger volume produces correspondingly more emissions, care must also be taken during the calibration process. In this study, all of the specimens have the same volume.

Figure 9 shows the acoustic emission response of a sample oven-aged for 36 hours and of a sample aged for 2-hours, i.e., short-term aging, respectively, when cooled from room temperature to -35$^{o}$C. Following our definition of embrittlement temperature, Fig. 9 shows the embrittlement temperatures of the oven-aged specimen for 36 hours and for the short-term aged specimen to be around -15$^{o}$C and -25$^{o}$C, respectively. These embrittlement temperatures are consistent with those quantitatively evaluated based upon the binder's rheological properties

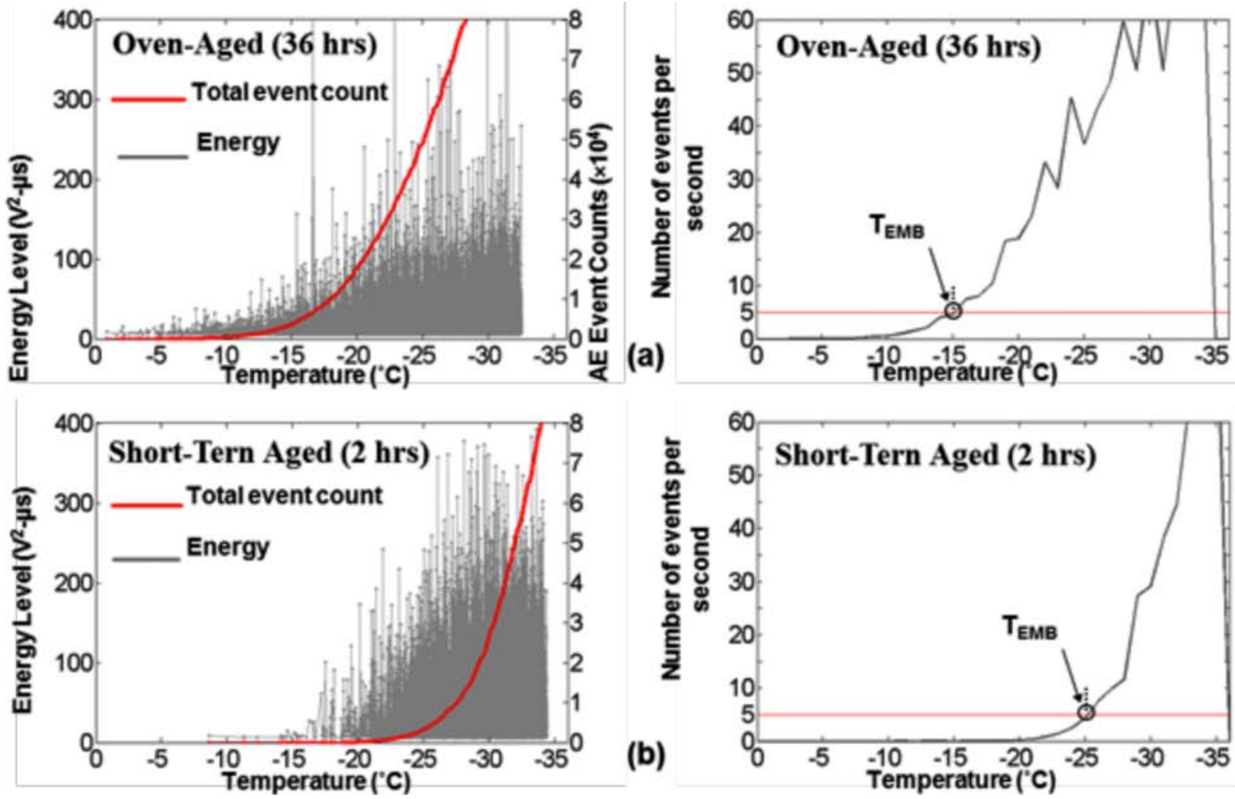


Fig. 9. The first and second row represent AE tests results for asphalt concrete samples oven-aged for 36-hours and for 2-hours, respectively. Left column represents AE event energy levels and AE total event counts versus temperature for asphalt samples observed during cooling. Right column represents the average event count rate versus cooling temperature, and shows the corresponding embrittlement temperatures.

(AASHTO MP1, 1998; AASHTO TP1, 1999; AASHTO MP1A, 2001). Figure 9 shows that the aging of the binder results in a sample with warmer embrittlement temperature of -15$^{o}$C rather than -25$^{o}$C. Figure 9 also shows that the short-term aged sample, while having less AE events, also has events that are more energetic and occur at cooler temperatures than the AE events associated with the oven-aged specimen for 36 hours. This difference in acoustic emission response is consistent with the observation that the binder of the specimen aged for 36 hours is stiffer and has lower adhesive properties than the "control" binder, i.e., binder with short-term aging. Because of the oven–aged process, in addition to the binder becoming stiffer, micro-flaw populations also develop between the binder and the aggregates and between the binder and the mastic because of lower adhesive properties of the binder. This also explains why the embrittlement temperature of the specimen oven-aged for 36 hours is warmer than the embrittlement temperature of the specimen for short-term aging. During cooling the developed thermal stresses reach the binder fracture strength at warmer temperatures (because of the presence of the micro-flaw populations) as compared to the short-term aged specimen.

Figure 10 shows the acoustic emission response of the four test samples exposed to rejuvenator on the top surface after the dwell times of two, four, six, and eight weeks, respectively. The left column represents the average of the AE activity collected by four AE sensors coupled to asphalt surface not exposed to rejuvenator, i.e., bottom surface, and right column represents the average of the AE activity collected by four sensors coupled to the asphalt surface exposed to rejuvenator, i.e., the surface where the rejuvenator was applied or the top surface. The

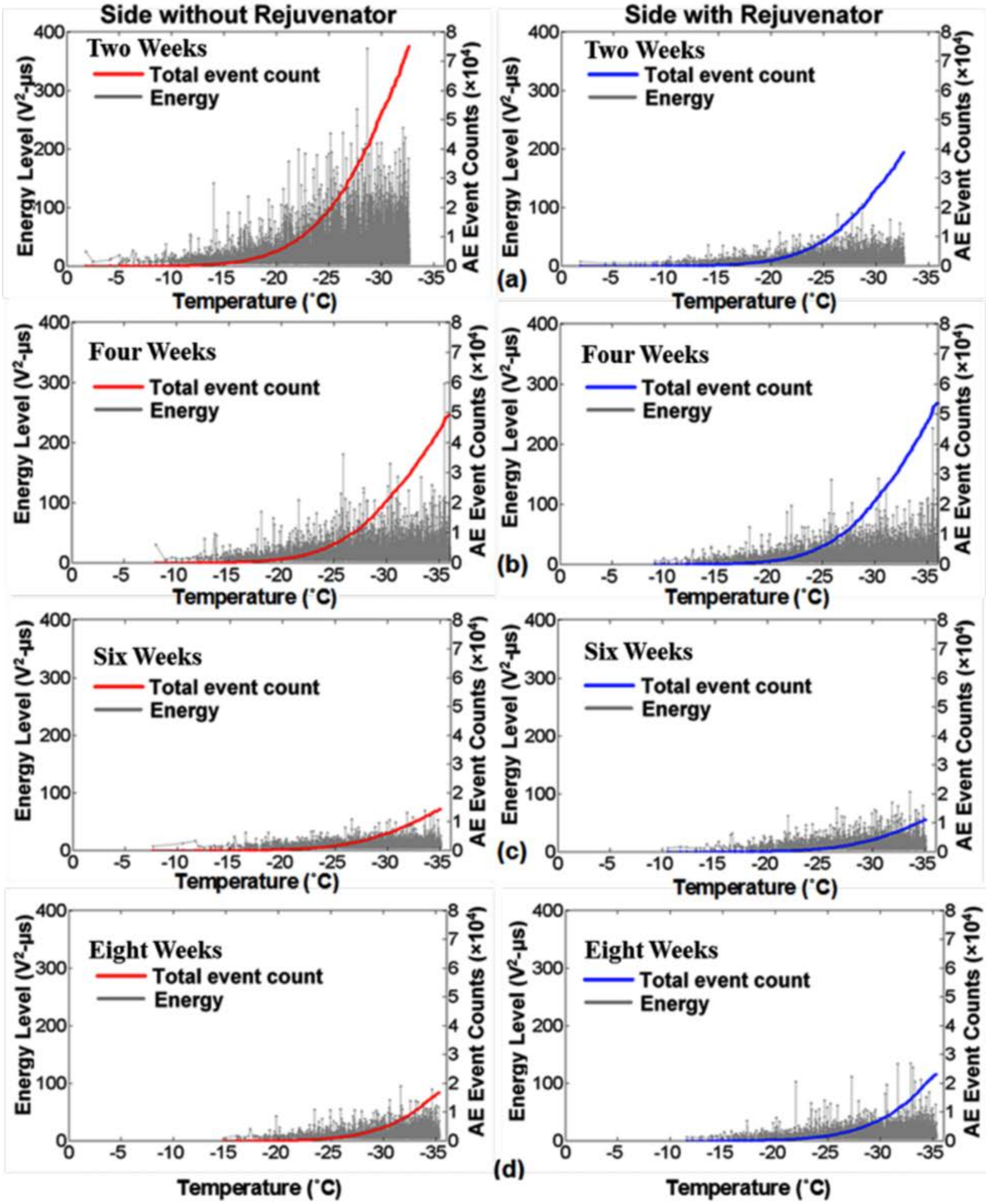


Fig. 10. AE event counts and AE event energy versus temperature for asphalt samples oven-aged for 36 hours and exposed to rejuvenator with a dwell time of (a) two weeks, (b) four weeks, (c) six weeks and (d) eight weeks. Left column represents the average of four sensors coupled to the asphalt surface not exposed to rejuvenator. Right column represents the average of four sensors coupled to the asphalt surface exposed to rejuvenator, i.e., surface where the rejuvenator was applied.

rejuvenator penetrates the specimen via gravity and capillary action. Once the rejuvenator contacts the binder it acts on the binder making it more viscoelastic as compared to the oven-aged binder for 36 hours. Figure 10 shows that after only two weeks of dwell time the surface exposed to rejuvenator has significant less acoustic emission activity than the bottom surface, i.e., the surface not exposed to the rejuvenator, which is similar to the AE response of the specimen aged for 36 hours without rejuvenator, see Fig. 9. As the dwell time increases and the rejuvenator has the time to reach the bottom of the specimen and act upon the binder, similar emission activities are observed by the sensors coupled to both the specimens' top and bottom flat surfaces.

To estimate the effect of rejuvenator upon the embrittlement temperatures, Fig. 11 shows the number of events per second versus the cooling temperature. The left column represents the average of AE activity collected by four sensors coupled to asphalt surface not exposed to rejuvenator, i.e., bottom surface, and right column represents the average AE activity by four sensors coupled to asphalt surface exposed to rejuvenator, i.e., top surface. Figure 11 shows that as the dwell time increases, and the rejuvenator has time to act on the oven-aged binder, the embrittlement temperatures of the specimens become cooler, i.e., improve. Figure 11 shows that just after four weeks of dwell time the embrittlement temperatures quantitatively evaluated from data provided by AE sensors coupled to both surfaces reached the embrittlement temperatures of the virgin specimen, i.e., the original embrittlement temperatures were recuperated. Figure 11 also shows that as the dwell time increases, the embrittlement temperatures of the specimens become cooler than the embrittlement temperatures associated with the short-term aged specimen. Please note that, while much care was exercised during the manufacturing of the test specimens, the variability observed in Figs. 10 and 11 is due to the inherent variability of material properties in the specimens such as porosity, aggregate distribution, level of compaction, etc.

## 4. Conclusions

An acoustic emission approach has been developed to quantitatively evaluate the embrittlement temperatures of asphalt concrete mixtures by monitoring the acoustic emission response of the test specimens during cooling. Acoustic emission tests were performed on short-term aged asphalt concrete samples as well as asphalt concrete samples oven-aged for 36 hours at 135$^o$C. It was observed that the embrittlement temperatures of the short-term aged specimens and specimens oven-aged for 36 hours were -25$^o$C and -15$^o$C, respectively. Four specimens oven-aged for 36 hours were then treated with rejuvenator (10% by binder weight) by spreading rejuvenator on the specimens' top surface. Using the same acoustic emission approach, the embrittlement temperatures were also evaluated after two, four, six, and eight weeks of rejuvenation application (or dwell time, i.e., time to allow rejuvenator to diffuse into the specimen and act upon the binder) on the asphalt concrete samples oven-aged for 36 hours. It was observed that during the cooling process, the short-term aged specimens have a lower number of acoustic emission events and more events of higher energy than the specimens oven-aged for 36 hours. This observation is consistent with previous observations that as the binder ages it becomes stiffer, with reduced adhesive properties. It was also observed that after four weeks of dwell time, the rejuvenator-treated samples have recuperated the original embrittlement temperatures. In addition, it was also observed that the rejuvenator kept acting upon the binder after four weeks of dwell time; at eight weeks of dwell time, the specimens had an embrittlement temperature about one grade cooler than the embrittlement temperature corresponding to the short-term aged specimen, where "one grade cooler" denotes "6$^o$C cooler." Based on this experiment, it appears that the proposed acoustic emission approach provides a method to evaluate the efficiency of rejuvenators to recuperate the embrittlement temperatures caused by oxidative aging of asphalt concrete mixtures.

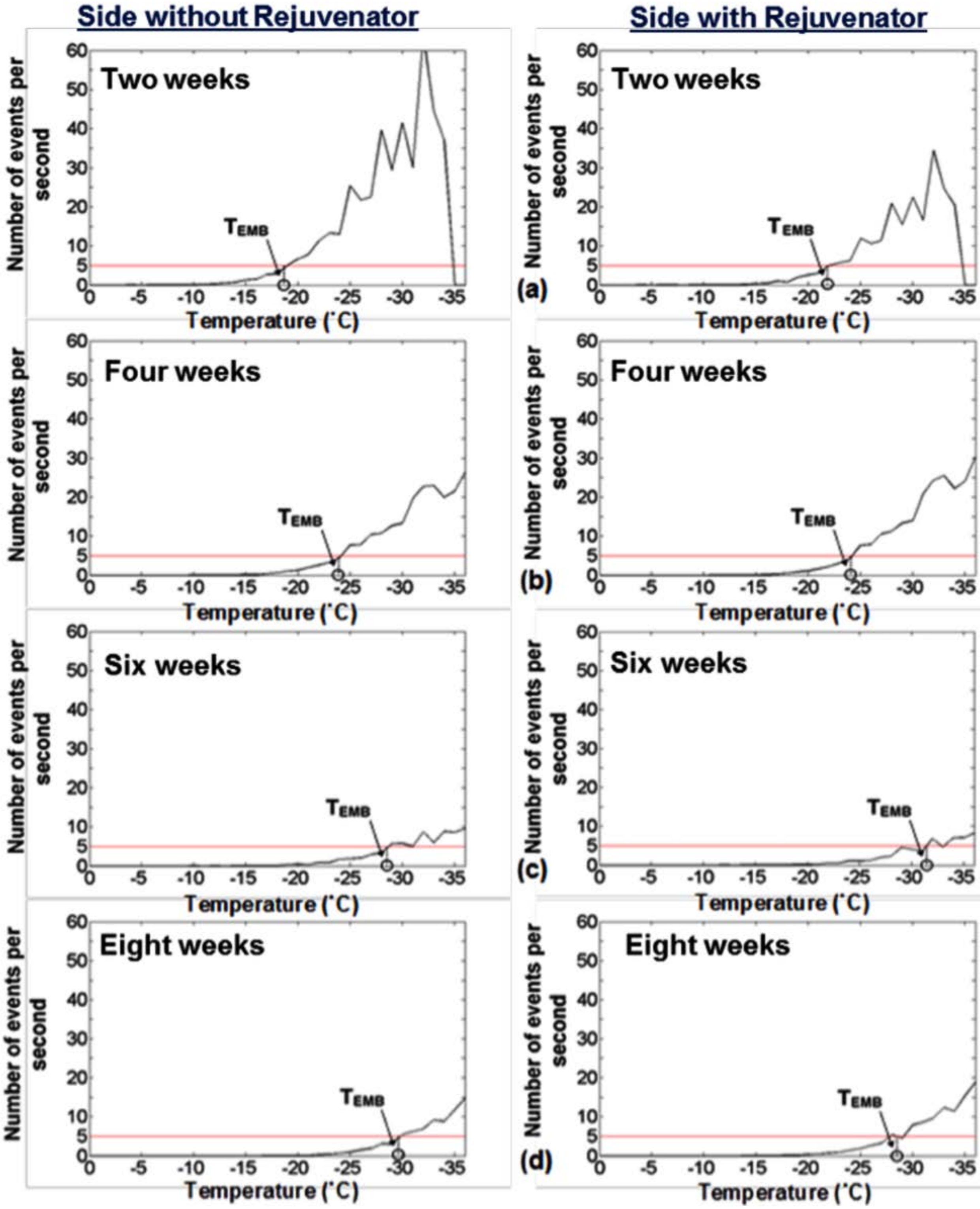

Fig. 11. Average rate increase in AE event counts versus cooling temperature for asphalt concrete samples oven-aged for 36 hours and exposed to rejuvenator with a dwell time of (a) two weeks, (b) four weeks, (c) six weeks and (d) eight weeks, and the corresponding embrittlement temperatures. Left column represents the average of four sensors coupled to asphalt surface not exposed to rejuvenator. Right column represents the average of four sensors coupled to asphalt surface exposed to rejuvenator. The amount of rejuvenator was 10% of the binder by weight.

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